

1981

# Some Physical and Chemical Properties of Pine Bark Growing Medium Used as an Evaluation of Its Nutritional Status.

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SOME PHYSICAL AND CHEMICAL PROPERTIES OF PINE BARK  
GROWING MEDIUM USED AS AN EVALUATION OF ITS NUTRITIONAL  
STATUS

The Louisiana State University and Agricultural and Mechanical Col.      PH.D. 1981

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SOME PHYSICAL AND CHEMICAL  
PROPERTIES OF PINE BARK GROWING  
MEDIUM USED AS AN EVALUATION  
OF ITS NUTRITIONAL STATUS

A Dissertation

Submitted to the Graduate Faculty of the  
Louisiana State University and  
Agricultural and Mechanical College  
in partial fulfillment of the  
requirements for the degree of  
Doctor of Philosophy

in

The Department of Horticulture

by

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December 1981

#### ACKNOWLEDGEMENT

The author wishes to express sincere appreciation to Dr. E. N. O'Rourke for his guidance, assistance and support in conducting this study. Gratitude is also expressed to Dr. D. W. Newsom, Dr. J. E. Sedberry, Jr., Dr. K. L. Koonce and Dr. E. P. Dunigan for serving as committee members and for their review of the manuscript.

The author also wishes to express his thanks to Mr. S. J. Guedry, Jr., Mr. R. E. Henderson and Mr. R. H. Brupbacher for their valuable assistance in collecting and analyzing samples.

Special appreciation is expressed to the author's parents, Mr. and Mrs. Don S. Wilkerson, for their encouragement and support throughout this study.

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## ABSTRACT

Various physical and chemical analyses were conducted on southern pine bark. The results from these analyses were then examined in terms of their effect on the interpretation of substrate fertility.

The drainage aeration and water holding capacity of pine bark was found to be suitable for use as a container medium. Also the determination of the pH, CEC, and "available" nutrient content of pine bark indicated acceptable levels for plant growth. However, all values were dependent on the method of analysis.

In general, it was concluded that a complex relationship exists between the physical and chemical properties of pine bark. Therefore, substrate fertility must be evaluated in terms of this interaction.

Foliar analysis was conducted on Euphorbia pulcherrima Willd. cv 'Annette Hegg Dark Red' grown on a constant fertilization program. The elemental content of the bark growing medium from these plants was assessed by three modified analytical methods. Probable optimum values were then determined for each of the three methods based on plant uptake.

These estimated nutrient levels varied according to the type of analytical system used. In general, exchange extractants yielded greater values than bulk solution extractants. However, each were effectively related to plant growth.

## INTRODUCTION

During the 1950's a great deal of attention focused on the use of "soil mixtures" for the production of containerized crops. These growing media generally combined soil with amendments such as sand and peatmoss to improve drainage, aeration and water-holding capacities. Gradually, these soil mixtures have developed into the "soilless" growing media which have come to dominate the floricultural scene.

Southern pine bark constitutes one of the largest sources of soilless growing media used today. Its coarse texture and relatively high moisture-holding characteristics make pine bark an excellent material for use as a container medium.

Frequently samples of bark media are submitted to the Louisiana State Soil Testing Laboratory for analysis. Although these samples can be put through the routine analytical procedure, the results are difficult, if not impossible, to interpret.

In 1979, the LSU Department of Horticulture undertook a study, in cooperation with the LSU Soil Testing Laboratory, to investigate the problems associated with the analysis of

pine bark growing media. Two basic objectives were established: 1) To develop a modified analytical procedure which could be integrated with methodology currently in use; and 2) To demonstrate that the values obtained from the modified analytical procedure could be related to plant response.

Fulfillment of these objectives will provide a means of assessing levels of fertility in pine bark growing media and serve as a guide for the interpretation of analytical results from other sources of soilless growing media.

## REVIEW OF LITERATURE

### Description and Origin of Southern Pine Bark:

The production of containerized floricultural crops presents several cultural problems. Because of its many facets, perhaps the most important of these is substrate management. The physical properties of the rhizosphere and growing media are significantly altered by the shallow depth and limited volume of a container. Therefore, special attention must be given to the basic physical and chemical properties of the growing medium if plant growth is to be maintained at optimum (132).

Most field soils generally have unsuitable drainage, aeration and water-holding characteristics for use in containers. To improve this situation, several soilless growing media have been developed. Tree barks, a primary by-product of the pulp, paper and plywood industries, constitute a major source of these soilless media. When hammer milled and screened, this material possesses many of the essential characteristics necessary for container production.

Soft woods make up the primary type of bark utilized in the preparation of these growing media. Among this group are a number of species indigenous to the southern United



States. These "southern" pines have been defined as those species whose major range is south of the Mason-Dixon line (lat. 39° 43' N) and east of the great plains (233). All of these species have at least fifty percent of their standing volume in the South. Combined, the southern pines occur on more than one hundred million acres of commercial forest land (219). All pines belonging to this group are diploxy-lon members of the order Coniferales, family Pinaceae, genus *Pinus* (Table I).

Table I. Southern Pines of the United States.

---

<u><i>Pinus palustris</i></u> Mill.	Longleaf Pine
<u><i>P. elliotii</i></u> 'elliotii' Engelm.	Slash Pine
<u><i>P. elliotii</i></u> 'densa' Little & Dorman	S. Fla. Slash Pine
<u><i>P. taeda</i></u> L.	Loblolly Pine
<u><i>P. echinata</i></u> Mill.	Shortleaf Pine
<u><i>P. glabra</i></u> Walt.	Spruce Pine
<u><i>P. virginiana</i></u> Mill.	Virginia Pine
<u><i>P. clausa</i></u> (chapm) Vasey.	Sand Pine
<u><i>P. rigida</i></u> Mill.	Pitch Pine
<u><i>P. erotina</i></u> Michx.	Pond Pine
<u><i>P. pungens</i></u> Lamb.	Table Mountain Pine

---

The four major southern pines, loblolly, shortleaf, longleaf and slash make up approximately ninety percent of the total southern pine inventory. The growth characteristics, range

and volume of these species strongly influences their availability and utilization in specific areas.

The loblolly pine (Pinus taeda L.) is the principal commercial pine species in the southern U. S. and accounts for nearly one-half of the total southern pine inventory. These trees thrive on soils with poor surface drainage, a deep surface layer and firm subsoil. Loblolly pines grow at elevations ranging from sea level to greater than two thousand feet in northern Alabama and Georgia. Rainfall in the growth range of the loblolly averages forty to sixty inches and temperatures from -10°F to over 100°F. Individual trees may attain diameters of fifty inches with a mean annual growth rate of 1.6 inches d.b.h. (diameter breast height), depending on stand management and environmental conditions (242).

The shortleaf pine (Pinus echinata Mill.) is the second most abundant southern pine species in the U. S. It has the widest range of the principal species and may be found in twenty-two different states. These trees prefer fine, sandy or silt loam soils without a distinct profile but with good internal drainage. Shortleaf pines may be found growing at elevations from ten feet to thirty three thousand feet above sea level. Within the shortleaf region mean temperatures range from 26°F to 80°F. In Oklahoma and Texas the forty inch annual precipitation line marks the southwestern boundary of the range. On good sites shortleaf pines attain heights of eighty to one hundred feet and diameters of two to three feet. Mean height growth

ranges from 2.3 to 2.8 feet per year (232).

The longleaf pine (Pinus palustris Mill) ranks after loblolly and shortleaf in abundance and commercial importance. These trees grow best on soils which are low in organic matter, sandy in the surface portion and well drained. Although longleaf pine may be found at elevations greater than two thousand feet, they usually grow at low altitudes. Annual precipitation exceeds fifty inches in much of the longleaf region and nowhere falls below forty inches. Average annual temperature ranges from 60°F to 70°F with a mean winter temperature of 45°F. Longleaf pines are relatively small trees with an average diameter of twenty five inches and rarely exceeding eighty feet in height. The mean growth rate is approximately one to three feet per year.

The slash pine (Pinus elliotii Engelm.) is the least widespread of the four principal southern pines. These trees grow most typically on sandy soils, sometimes having poorly drained hardpans eighteen to twenty-four inches below the surface. The natural region of the slash pine is characterized by an average annual precipitation rate of fifty inches, with an average annual temperature of 53°F. Topography and elevation vary only slightly within its limited area. Individual trees may attain heights of one hundred feet and diameters exceeding ten inches (215).

#### The Formation and Anatomy of Southern Pine Bark:

Bark formation in southern pine is dependent upon the seasonal activity of the vascular cambium. Each growing

season the cambium forms a layer of bark that is one-tenth to one-sixth as thick as the layer of wood formed. The new layers of inner bark, or phloem, push the older, non-functional phloem outward with expanding girth. These two phloem layers are separated by the protective periderm which is produced from actively dividing pelloge, or cork cambium, cells. Since outer layers of nonfunctional phloem and periderm are not sloughed off as rapidly as functional interior ones are formed, the thick, scaly bark, typical of southern pines, is accumulated.

The vascular system of the southern pines is typical of that found in many of the gymnosperms. This includes the absence of vessels from the xylem, and companion cells from the phloem. Also sieve cells replace the sieve tube members which are characteristic of the angiosperms (240).

The layer of tissue from the vascular cambium outward to the periderm, referred to as inner bark, contains the phloem produced during a number of years. This layer ranges in thickness from 0.5 mm to approximately 2.0 mm (103). However, only a very narrow band (approximately 200-300  $\mu$ m) next to the cambium is active in the translocation of photosynthetic products (105). The primaries of these food conducting elements are the vertically oriented sieve cells. The thin, nonlignified, cellulosic walls of these sieve cells have numerous circular to oval sieve areas. These vary in size and number of pores according to cell dimension and position. The functioning life of a sieve cell is dependent on a carbohydrate deposition referred to as callose.

With reduced activity of the cell, callose grows to become a massive accumulation. This generally marks the functional end of the sieve cell. While still in the inner bark layer, these sieve cells lose turgidity, collapse, and their orientation becomes distorted. It has been estimated that this process occurs one to two years after formation (216). Sieve cells constitute approximately fifty percent of the inner bark and less than thirty percent of the outer bark layer, or rhytidome. This reduction is attributed to cellular collapse, parenchyma enlargement and the introduction of periderm tissue.

The principal function of parenchyma cells is the storage of water and other products. Within the inner bark layer, longitudinal parenchyma may be found dispersed among the sieve cells. Because tangential arrangement of parenchyma cells is soon distorted, it is generally not observed in the rhytidome. Primary pit fields in the walls of parenchyma cells permit communication with other parenchyma and ray cells. However, parenchyma cells usually do not appear to be associated with sieve cells (217, 167). Starch, tannins, resins, oil globules, and crystals are among the primary substances stored in phloem parenchyma.

Phloem rays are continuous with xylem rays and perform similar conductive functions. Ray cells associated with phloem have thin, primary, un lignified walls. Although no tracheids are present, erect structures referred to as albuminous cells form the margins of most rays. These cells are physiologically associated with sieve cells and are

thought to regulate cellular activities and serve in translocation between sieve cells and ray parenchyma. Albuminous cells die and collapse simultaneously with their associated sieve cells. Except for those albuminous cells, rays remain alive throughout inner bark and transport nutrients from the actively conducting zone to living longitudinal parenchyma and the innermost phellogen. The outermost portions or rays die when ultimately sealed off by the formation of a new and deeper phellogen.

Crystals, composed of calcium oxalate, occur within both the inner bark and rhytidome. These crystals are deposited in the lumina of sieve cells and longitudinal parenchyma as metabolic by-products (237, 115). Therefore, the quantity of these crystals in a particular sample may be dependent on nutrient availability and tree vigor (83).

The transformation from inner bark to rhytidome involves: the formation of the periderm; the alteration of cell structures and arrangements; and the deposition of substances within the cells.

As sieve cells reach their functional end, vertical parenchyma cells enlarge. This process, referred to as obliteration, has been shown to occur most readily in bark near ground level as opposed to bark high in the tree (186). In the inner bark layer parenchyma cells are largely outnumbered by sieve cells, however, in the rhytidome these enlarged parenchyma cells occupy most of the volume, creating a very porous structure.

Rhytidome contains abundant deposits of tannins,

phlobaphenes and other phenolic substances. Although the physiological function of these compounds is not clear, it has been suggested that they act as naturally occurring fungicides and antioxidants (122).

The inner bark is protected by the periderm which consists of phellogon and phellem cells. The phellogon, a single layer of cells, divides tangentially to produce radially aligned cells toward both the inside (phelloderm) and outside (phellem). The function of phelloderm is not entirely understood, and changes in its structure occur at various distances from the phellogon. In most species, the volume between periderms is occupied by expanded phelloderm. Phellem cells are primarily responsible for the protective characteristics of the periderm. Two distinct types of cells occur within the phellem region: thin-walled cork cells and thin-walled stone cells. Cork cells have unpitted cellulose walls interlaid with a lamella of suberin and waxes, which render them practically impervious to moisture and gases (131, 80). These cork cells are compactly arranged without intercellular spaces. Some phellem cells sclerify and become stone cells. These are about the same size as cork cells but have very thick secondary walls with ramified pit canals. Stone cells form the only heavily lignified tissue in southern pine bark.

Several studies have been conducted on the basic surface and microstructure characteristics of the southern pine species (211, 47, 139). However, based on these criteria, no significant differences have been found between species.

The Physical Properties of Southern Pine Bark:

The wood content of trees is generally assessed by measuring circumferences outside the bark. Therefore, reliable means for estimating bark thickness have been developed (198, 5). Strong correlations have been shown to exist between bark thickness and diameter for the four major southern pines (41, 133, 110, 157). However, a more accurate assessment of in-bark volume takes into account the corrugated nature of the bark surface and the variability in bark thickness around the circumference of the tree (155).

Another means of estimating bark thickness is by determining the ratio between diameter at breast height and bark thickness. In one such study McCormack (145) reported that for every inch in breast height diameter growth of inside bark, the diameter of outside bark increased according to specific ratios (Table II).

Table II. Ratio between growth of inside and outside bark for the four major southern pine species.<sup>1</sup>

Pine species	Ratio
Loblolly	1.10
Shortleaf	1.09
Longleaf	1.06
Slash	1.07

Variation in the moisture content of southern pine bark is primarily accounted for by differences between inner and



outer bark. The moisture content of inner bark ranges from 150 to 300 percent (oven dry), while outer bark ranges from 18 to 63 percent (143, 191). Because the innermost periderm separating these two layers is relatively impervious to water, the transition in moisture content from inner to outer bark is abrupt (92). Therefore, the moisture content of southern pine bark is largely dependent on the ratio of outer bark to total bark. Snow (213) has shown that outer bark constitutes approximately 75 percent of the total bark weight when freshly cut. Further study has shown that the moisture content of whole stem bark ranges from 80 to 100 percent. Other factors, such as seasonal changes, diameter at breast height, height above ground, and age of tree have also been found to influence moisture content (103).

Variation in the specific gravity of southern pine bark occurs among species, among trees within the same species, and within individual trees (140). Much of this variation is attributed to the expansion of old phloem cells and the distribution of phellem stone cells. Although data are insufficient to permit definition of species averages, the specific gravity of the outer bark of the four major southern pine species has been estimated (Table III).

Several studies (141, 142, 346) have been conducted to determine the source of variation in specific gravity. Factors such as tree age and vigor, diameter at breast height and height above ground have all been investigated. However, conclusive evidence has not yet been presented which supports any or all of these hypotheses.

Table III. The specific gravity of the four major pine species.

Pine species	Specific Gravity g/cc	Standard Deviation
Loblolly	.477 <sup>1</sup>	.105
Shortleaf	.473	.106
Longleaf	.640	.097
Slash	.474	.085

Studies regarding the volumetric expansion of southern pine bark have indicated that volume changes do not occur at moisture contents above the point of fiber saturation (119). It has also been shown that shrinkage between saturation and the oven dry condition is linear (37). Based on this information the volume of a piece of bark may be computed at any moisture content given the wet ( $V_w$ ) or dry ( $V_o$ ) volume:

$$\text{where: } V_m = (0.8647 + 0.00534 M) (V_w)$$

$$V_m = (1 + 0.00618 M) (V_o)$$

M = moisture content as percent of  
the oven-dry weight

#### The Chemical Properties of Southern Pine Bark:

Most of the work which has been conducted on the chemical constituents of southern pine bark has not been approached from the standpoint of a growing medium. For wood utilization purposes most interest centers around rhytidome after its removal from mature trees (100, 52). The general analysis of whole southern pine bark yields five

classes of chemicals: ash, hollocellulose, lignin, phenolic acids and extractives. Although the effect of each of these on plant growth is unclear, their presence creates a set of chemical properties unique to southern pine bark.

The occurrence of carbohydrates in southern pine bark is important in terms of microbial activity and decomposition. The carbohydrate content of slash pine bark, as determined by hydrolysis with 72% sulfuric acid, was shown by Chang and Mitchell (48) to yield reducing sugars (measured as glucose), ranging from 26 to 30 percent. Browning and Sell (38) further reported the percent of sugars in the hydrolysate of extracted bark as 63% glucose, 15% xylose, and 7% each of galactose, mannose, and arabinose.

Several studies (65, 27) have been conducted which characterize the properties of lignin in southern pine bark. This work has shown average lignin content ranging from 45 to 50 percent in extractive-free, oven dry pine bark. This lignin fraction consists of bark lignin plus insoluble corky substances from the acid insoluble residue (126). There has also been evidence presented which supports the presence of a lignin-carbohydrate linkage in bark that is absent in wood (126).

Perhaps more work has been conducted on phenolic acids and extractives than any other phase of southern pine bark chemistry. However, little is known of their origin or function in the plant (98, 70, 218, 104). These two closely related groups of chemical compounds are composed of a number of various substances: tannins, resins, waxes,

saponins, starches, simple carbohydrates, alkaloids and minerals (86); which are primarily used in the identification of various pine species.

The ash content of southern pine bark has been shown to constitute approximately 0.6 percent of its oven dry weight (156, 92). However, variation within and between trees has been significant, with increasing ash content occurring in the upper crown of the tree.

#### The Preparation of Soilless Growing Media from Southern Pine Bark:

Considerable research efforts have been directed toward the evaluation of southern pine bark as a growing and propagation medium (55, 62, 79, 179). The performance of bark in these applications is largely influenced by the debarking and grinding process. Several types of bark removing equipment are now in commercial operation and each has its own effect on the quality of growing media which may be produced.

Ring and drum debarkers remove less than 10 percent wood from logs and generally yield bark with excellent media potential. Although Rosserhead and Hosmerhead debarkers remove considerably more wood from logs, they are also considered suitable for use in a container media operation (101). The paper industry has recently introduced whole tree chippers which may remove bark with up to 60 percent wood. This type of debarking process is not generally considered suitable for media use (194).

After removal, bark is hammermilled and screened. The resulting distribution of small, medium and large particles

will determine such factors as percent pore space, drainage and moisture-holding capacity. Although no formula is available which predicts such properties, a uniform texture usually results (136).

Of primary concern with any organic growing medium is the effect of its carbon to nitrogen ratio (C:N) on the loss of nitrogen to micro-organisms using the organic matter as a carbon source. Several studies (18, 109) have been conducted on the C:N of pine bark growing media and its effect on plant growth. However, due to the relatively short growing period and high fertility regimes used for most floricultural crops, this is not a significant problem (17). A practical means of handling nitrogen depletion is by establishing partially controlled conditions for decomposition prior to media preparation. This "composting" process provides optimum conditions for accelerated decomposition until the physical and chemical properties of the bark have been adjusted to those desired (3, 177). Many growers feel that composting produces a higher quality of bark for use as a growing media. However, there are those who believe that excessive composting decreases the disease-suppressing qualities associated with pine bark.

#### The Physical Analysis of Soils and Soilless Growing Media:

The precise control of water and fertilization is necessary to maintain optimum growth in containerized floricultural crops. Therefore, detailed knowledge of the physical properties of the growing medium is of prime importance (58).

The bulk density of a growing medium has been defined as the weight of solids per unit volume of total media (oven dry) (227). Although any properly specified units may be used, bulk density is usually expressed as grams per cubic centimeter (g/cc).

Particle size distribution, specific gravity and compaction are all factors which affect the bulk density of a growing medium (24). However, most variation in bulk density arises from differences in percent total pore space. Generally as percent total pore space increases, bulk density decreases (61). However, percent total pore space may be calculated as a function of bulk density and specific gravity where:

$$\% \text{ total pore space} = (1 - \text{BD}/\text{specific gravity}) \times 100$$

Therefore, this axiom may also be stated in terms of bulk density (as bulk density increases, percent total pore space decreases).

The compaction of soilless growing media results from initial pressure applied during potting and by settling following subsequent irrigation. These two factors have been shown to affect bulk density in both soils and soilless growing media (223).

Pokorny (178) has reported bulk density values for southern pine bark ranging from 0.2 to 1.56 g/cc. However, it is difficult to interpret these values without some indication of particle size distribution. The particle size distribution of a growing medium is determined by means of

U. S. Standard Sieves, with values usually expressed as a percent of the total sample weight (26). To a large extent the particle size distribution or texture of a pine bark growing medium is predetermined by the screening process during media preparation. However, when a mixture of soilless components is used in the synthesis of a growing medium (i.e., sand and bark), a determination of particle size distribution, on a weight basis, may underestimate a particular fraction. Such variation is attributed to differences in the specific gravity of media components. In such cases a volume basis is recommended for the determination of particle size distribution (229).

The particle density of a growing medium is defined as the average density of its particles (225). Like bulk density, values for particle density are expressed in units of weight per volume, usually grams per cubic centimeter.

Blake (15) has described the principal method used for the determination of particle density in soils. He points out that most variation from this method results from inaccurate volume and weight measurements. For example, a weighing error of 10 mg in a 30 g sample can give a particle density error of 0.001 g/cc. A 0.2 ml volume error on a 40 g sample can result in a compounded particle density error of 0.15 g/cc. In addition to weight and volume errors, it must also be assumed that some random error exists.

Gradwell (81) has shown that soil particle density values are generally 0.01 to 0.03 g/cc higher in water than in nonpolar liquids (i.e., toluene, xylene or carbon tetra-

chloride). This is also dependent on the adsorption activity of the soil being tested. Therefore, the use of specialized liquids may be necessary for accurate determinations (4, 234).

The amount of water held by an air-dried growing medium, referred to as capillary water (227), may be calculated as a percentage of the oven-dry weight:

$$\% \text{ capillary water} = [(W_1 - W_2)/W_2] \times 100$$

where:  $W_1$  = weight with capillary water

$W_2$  = oven-dry weight

In many chemical and physical analyses a separate capillary moisture determination is required. Although capillary moisture may vary with atmospheric pressure and humidity the primary source of variation is the structure of media components. Airhart, Natarella and Pokorny (1) have studied the internal structure and external surfaces of processed pine bark in relation to moisture retention. Their findings have shown numerous cracks and openings as well as internal cellular connections which might allow water penetration. However, they have also shown the existence of several waxy, suberized layers which may contribute to the hydrophobic character of dry pine bark. It has also been shown that some diethyl ether and methanol extractable substances interfere with water penetration. Furthermore, it has been hypothesized that the size of the openings might also be part of the difficulty in wetting (34).

The moisture content of a soil (in the absence of evaporation) when downward movement of water has virtually



ceased is referred to as field capacity (227). A comparable measurement for growing media used in containers has been proposed by White (244). He defines "container capacity" as the total percent water, by volume, held by a growing medium in a container of a given depth with zero hydraulic head (saturation), at its lower surface and in the absence of evapotranspiration. Container capacity is closely related to particle size distribution, and is influenced by media components and their structure.

The rate at which water moves through a growing medium is referred to as its percolation rate (227). For most applications with soilless growing media, percolation rate is expressed in centimeters per fifteen minutes; however, this may vary. Since most soilless growing media are designed to provide good drainage, they are generally prepared from coarse materials. Therefore, it is necessary to utilize a constant hydraulic head permeameter for the percolation rate determination:

$$\text{Percolation rate} = Ql/TAh$$

where: Q = the volume of water

T = time

A - cross-sectional area

l - length

h - loss in hydraulic head through l

Fireman (73) has described the constant-head method most frequently used for such determinations. No differences in percolation rate have been found upon varying the

length of the permeameter from 1 to 34 inches, or diameter from 1 to 6 inches. However, significant variation may result from differences in compaction and particle size distribution. Therefore, uniform samples are necessary for accurate analysis (16).

Maintaining a constant hydraulic head is important and several methods of water-level control have been developed (241). However, if operated properly, there is no advantage (except convenience) to any particular system.

The percolation rate of a porous medium is presumably a constant and independent of the fluid used in the determination (95). However, it has been shown that the presence of hydrophilic colloids precludes the use of anything but water in the measurement of the percolation rate of a growing medium (74). Other factors such as viscosity, temperature, volume, weight, water content at packing, electrolyte concentration and hydraulic gradient also influence the determination of the percolation rate of a soilless medium (193, 50, 84).

#### The Chemical Analysis of Soils and Soilless Growing Media:

Soilless growing media may be considered as a practical extension of the complex analytical system of soils. These media are primarily designed to provide the optimum physical characteristics necessary for plant growth, without sacrificing those chemical properties associated with soil (144).

Although several analytical methods may be used to evaluate the efficiency of soils for plant growth, complications arise when these methods are applied to soilless

growing media. To obtain meaningful values from soil tests conducted on soilless growing media, adaptations of methodology are required. However, before such adaptations may be recommended, it is important to understand the basic principles and theories of the chemical analysis of soils and soilless growing media.

Sampling is perhaps the most fundamental soil test procedure. Although the Association of Official Agricultural Chemists (161) has studied this problem in detail, it has concluded that, in view of the variability of soils, no entirely satisfactory method of sampling is possible. Generally, the sources of sampling variability are divided into two categories: 1) soil variability and 2) the various types of analysis to be performed on samples. Therefore, sampling technique must not only take into account inherent variability within the soil, but handling, processing and subsampling as well (108).

The concept of volume is important in conducting and interpreting the results of soil tests (149). Samples taken from the sampling volume are used to make inferences pertaining to the entire soil body. For most applications the sampling volume of soils is the furrow slice (170).

The preparation of soil samples for analysis is also a function of the sampling technique. Soil samples are usually ground to pass through a 2 mm screen prior to analysis. This process breaks up aggregates and reduces particle size variation. Jackson (108) has recommended that

the largest particle of a soil sample should constitute 0.001, or less, of the minimum sample volume. Although most particles will be much larger, this will produce an optimum sample volume three to four times the minimum and reduce particle size variability.

After grinding, most soil samples are dried at temperatures ranging from 25°C-35°C and relative humidities of 20-60 percent (7). However, due to changes which occur in some ionic species upon drying, it is recommended that many types of analysis be conducted on freshly collected, moist samples. Examples of these include: exchangeable ferrous iron (89), hydrogen ion activity (228), exchangeable potassium (90), and nitrate nitrogen (12).

Profile and subsoil sampling are also very important factors in obtaining meaningful values from various strata of the soil (49). These sampling techniques take into account factors such as toposequence, climosequence, chromosequence and lithosequence (42).

Much of the variability in soil sampling may be controlled by the use of composite samples. Analyses conducted on composite samples result in mean values which represent those of the sampling volume (197). To a large extent composite samples reduce variability from the media sampled. However, analytical variation may also exist. Three principal sources of error have been associated with the various types of analysis performed on soil samples (85): variability among different samples drawn from the same volume (sampling error); variability introduced among subsamples

of the same sample (subsampling error); and variability from one determination to another on the same subsample (analytical error). Jackson (108) has shown that variation arising from sampling and sample treatment is significantly greater than variation from subsampling and analysis. Therefore, the reliability of the initial sample is most important in obtaining accurate results from analytical determinations.

Acids, when mixed with water, dissociate or ionize into hydrogen ions and associated anions. The stronger the acid the greater the amount of ionization. Those hydrogen ions which dissociate are measured as active acidity, while those capable of dissociating are measured as potential acidity. The sum of the concentrations of active and potential acidities yields total acidity (196).

In strong acids the activity of hydrogen ions is so nearly equal to the concentration of total acidity that there is little need for separate designations. However, many weak acids dissociate to less than one percent, in which case a measure of total acidity gives no indication of active acidity. With extremely weak acids, hydrogen ion activity is generally stated in terms of the logarithm of the reciprocal of hydrogen ion activity, or pH (227).

pH: Although Linsley and Bauer (129) have described a colorimetric method for the determination of soil pH, the most accurate and widely used method is by means of a glass electrode potentiometer. Glass electrodes are unaffected by oxidizing and reducing substances, and do not liberate

dissolved gases from the system (11). However, Reid and Cummings (190) have found that soil pH measurements may vary depending upon the method of sample preparation. The principal sources of this variation have been identified as drying, soil water content and soluble salt concentrations.

Soil pH measurements made under field moisture conditions may be considered the most valid in evaluating the existing soil environment (45). However, the drying process may hasten certain chemical reactions resulting in samples near equilibrium. The measurement of air-dried samples is the most frequently used procedure in the determination of soil pH (14).

Soil pH measured with a glass electrode potentiometer is conducted by placing a soil suspension in contact with the glass electrode. Since soil colloids behave as weak acids, the presence of a solid phase may be expected to give lower pH values when in intimate contact with the electrode. Conversely, as the soil suspension becomes more dilute, pH values tend to increase (106). Several dilution ratios have been recommended for the determination of soil pH. These range from the moisture saturation percentage to a 1:10 soil solution ratio (175, 112, 179). Although wider dilutions may be used, they generally require longer equilibration time (approximately 30 to 60 minutes) and must be read within 60 seconds of electrode immersion. Stirring the suspension is also necessary to keep the soil suspended during the pH measurement.

The accurate measurement of pH by the glass electrode

potentiometer requires an equal transfer of  $K^+$  and  $Cl^-$  ions from the salt bridge of the electrode (40). A significant liquid junction potential may exist if the diffusion of  $K^+$  ions exceeds that of  $Cl^-$  ions from the salt bridge. This would result in a potential difference above that from hydrogen ion activity. If the salt bridge comes into contact with the solid phase of the suspension an increased junction potential will occur. Therefore, thicker suspensions surrounding the electrode generally result in greater junction potentials and lower pH values (54).

To obtain an accurate evaluation of soil acidity soluble salts should be removed. The presence of salts influence ionic activities and, therefore, reduces pH values (189). This salt effect may be overcome by leaching with distilled water and then conducting pH determinations on the salt-free soil (172). Another method of masking the acidifying effects of soluble salts is to suspend the soil in a salt solution rather than water. Differences in soil pH caused by differences in the salt concentration of the soil solution will have no effect on the pH measured in the added soil-salt solution suspension. This is then a more precise evaluation of soil acidity than that measured in a soil-water suspension (51, 53, 201).

Exchangeable hydrogen: The determination of exchangeable hydrogen in soils is essentially an equilibrium determination of proton ( $H^+$ ) supplying power. When a strong proton acceptor is placed in equilibrium with the soil, protons are removed from it. This quantity of protons is

measured as exchangeable hydrogen (182).

Acid soils generally contain quantities of active or exchangeable aluminum (Al). When a strong proton acceptor is introduced to this system, hydrolysis and release of protons from water may result. The net effect of this is equivalent to the release of exchangeable hydrogen ions. The quantity of exchangeable hydrogen is, therefore, a function of the equilibrium system in which it is measured. Most determinations are conducted at pH values from 7.0 to 8.1 (247). Although several proton acceptors or extractants have been proposed for the determination of exchangeable hydrogen (107, 248, 183), the use of barium chloride, buffered at pH 8.1, is perhaps the most common method used (169).

Exchangeable aluminum: The effects of exchangeable Al upon soil acidity have been studied by Heddelson, McLean and Holawaychuck (97). Their findings have shown that as  $Al^{+3}$  ions, which are displaced by cations, hydrolyze in the soil solution the hydrolysis products are readsorbed causing further hydrolysis of  $H^+$  and hence lower pH.

The extraction of Al is a continuous function of soil pH, and with buffered solutions the pH of the extraction is standardized. With neutral salt solutions, the pH of the soil controls the extraction. If soils are approximately neutral in reaction the Al extracted by neutral salt solutions may be regarded as exchangeable. However, if the soil or extractant is acidic, the other forms of Al may be dissolved. Although several neutral extractants may be used



in the determination of exchangeable Al, potassium chloride is most common (127, 111, 147).

**Total Exchangeable Acidity:** The total exchangeable acidity of a soil is measured as the sum of exchangeable hydrogen and aluminum (35). If an unbuffered salt solution is used to replace the exchangeable hydrogen and aluminum from soil, it is possible to determine total exchangeable acidity in the leachate. If the aluminum in this solution is then converted to a stable complex, it too may be measured. The difference between total exchangeable acidity and exchangeable aluminum would then yield exchangeable hydrogen (250, 160).

Ion exchange is defined as the reversible process by which cations and anions are exchanged between solid and liquid phases. Although anion exchange is an important property, cation exchange is generally considered a more useful evaluation of soil fertility (96).

The solid phase of soils may be divided into the organic and inorganic fractions. Because cations are positively charged, they are attracted to negatively charged surfaces arising from COOH and OH groups in the organic fractions, and from isomorphous substitution and the broken edges of clay particles in the inorganic fraction (134). The negative charge which develops on organic and inorganic colloids is neutralized by cations attracted to their surfaces. The quantity of these cations (usually expressed in milliequivalents/100 g of soil) is then measured as the cation exchange capacity of the soil (152).

When a soil is extracted with a fairly concentrated, aqueous, salt solution, all of the adsorbed cations are replaced by ions from the extractant. If this saturated soil is then extracted with a salt solution having a greater affinity for adsorption, replacement of adsorbed ions will result. A determination of the quantity of these displaced ions may then be measured as the cation exchange capacity of the soil (203).

The cation exchange capacity of a soil is not a fixed quantity but is dependent on the pH of the extracting solution used (154, 205). When the cation exchange capacity of a soil is determined with an unbuffered neutral salt solution, the value obtained will be lower than when measured with a highly buffered solution (137). Therefore, the cation exchange capacity of a soil may be a rather arbitrary figure unless the method of determination is clearly stated (113).

Exchangeable cations in acid soils have been defined as those extracted with a neutral unbuffered salt (60). Such a solution will extract only the cations held at active exchange sites at that particular soil pH. The exchangeable acidity then extracted is  $A_1$ . The sum of the milliequivalents of basic cations and  $A_1$  has been termed the effective cation exchange capacity (59).

Several extractants may be used in the determination of the cation exchange capacity of soils (202, 153). The principal difference between these is the final method of determination of replaced ions in the leachate. Bauer (13) has described the method which is generally considered the

most rapid and simplified of all cation exchange determinations. Following this method, sodium acetate is employed as the index extractant, with final determination of sodium ions made by the convenient means of atomic adsorption.

**Base Saturation:** The total cation exchange capacity occupied by basic cations (i.e., Ca, Mg, K and Na) is defined as the percent base saturation of a soil (114). Base saturation may be related to both soil pH and level of soil fertility (165, 146). For a soil of any given organic and mineral composition, pH and fertility will increase with an increase in the degree of base saturation. Although several extractants have been suggested for the determination of percent base saturation (23, 209, 44), ammonium acetate is the most commonly used (202).

The exchangeable, often termed "available," cations which are of most frequent interest in soils include  $K^+$ ,  $Ca^{++}$ ,  $Mg^{++}$ ,  $Mn^{++}$  and  $Na^+$ . These cations may be readily replaced and their concentration determined in soil extracts. Selection of a particular extractant is dependent on its ability to extract cations that are correlated to plant growth.

Many of these extractants vary in their mode of action. Some measure the ionic composition of the soil by removing only bulk solution ions (dissolved soluble salts and soluble ions). These methods, however, do not remove ions from the exchange sites (9). Other types of extractants utilize dilute acids to simulate natural soil solution processes. These remove bulk solution ions as well as some ions from the exchange sites (160). Exchange extractants measure the

total ionic composition of the soil. This includes bulk solution ions as well as extensive displacement of ions from the exchange sites (23).

Soil:extractant ratios are very important in evaluating the quantity of exchangeable cations in soils. Although these may vary, depending on the soil being tested, several standardized methods have been devised.

Saturated soil pastes take into account textural differences between soils, allowing for variations in the amount of solution required by the soil to become saturated. Although errors in reproducibility may occur, this method is frequently used. Jackson (108) describes a saturated paste as being moist enough to glisten, slightly flowing, and capable of sliding off a spatula easily. He further notes that soils high in organic matter generally require soaking overnight before such a paste can be satisfactorily prepared.

The determination of exchangeable cations may also be conducted on extracts from the soil paste. These saturation extracts are generally obtained by suction, and the values obtained from these are dependent on the soil:extractant ratio of the original paste. Therefore, the percentage of solution used to prepare the saturated extract is needed for the interpretation of results (176). The saturation percentage may be calculated as follows:

$$\text{Saturation \%} = 100 \times (2.65 - W/2.65) (W - V)$$

where: V = volume of soil paste (cc)

W = the weight of V (g)

(It is assumed that 2.65 (g/cc) = the BD of soil)

In some cases exchangeable cation determinations on the actual soil solution are required. Several methods have been proposed for the extraction of soil solutions. The most obvious, suction, is often slow and is not always effective (150). Centrifuging can be used, but complications arise when samples are high in organic matter (32). Displacement is perhaps the most effective and frequently used method for soil solution extractions. In this procedure a displacing solution (i.e., ethanol) is allowed to percolate through a column of soil pushing before it the displaced soil solution. Subsequent determinations are then conducted on the leachate (29, 22, 212).

For most determinations of exchangeable cations, more dilute soil:extractant ratios are used. The selection of an approximate ratio is dependent on factors such as soil texture, pH, Ca-P concentrations, and interfering ions (88, 99, 163). Although these ratios are specific for individual analytical systems, generally values increase and become more stable as the soil:extractant ratio becomes wider (25).

The extraction time is also an important factor in determining concentrations of exchangeable cations. Although routine determinations rarely exceed thirty minutes, longer extraction times generally give rise to increased values which are more stable. However, this is dependent on the nature of the analysis, and to a large extent on soil pH, texture and interfering ions (162). The primary criterion for selecting an extraction time is its correlation with

plant response. Therefore, these factors will vary with soil-crop combinations (192, 226, 69).

Currently, several extraction systems are in use throughout the United States and the world. Although procedures are relatively similar, these systems utilize a variety of reagents.

The Spurway procedure (214) has long been accepted as the principal soil test method for horticultural crops. This system has been designed to simulate plant uptake and the solvent action is similar to that of water. The Spurway test employs 0.025 N acetic acid (HoAc) at a 1:4 soil:extractant ratio to remove bulk solution and some exchangeable ions from the soil (108).

Ammonium acetate ( $\text{NH}_4\text{OAc}$ ) is perhaps the most widely used exchange extractant in the U.S. for the determination of Ca, Mg, K and Na. It has been adapted to several soil test systems and is currently the extractant used by the L.S.U. soil test laboratory. This ammonium acetate system utilizes 1.0 N  $\text{NH}_4\text{OAc}$  at a 1:10 soil:extractant ratio to remove bulk solution and exchangeable ions from the soil (39).

The double acid soil test (151) is an extension of the ammonium acetate system. The double acid extractant combines reducing and oxidizing acids (0.05 N HCl and 0.025 N  $\text{H}_2\text{SO}_4$ , respectively) in a 1:4 soil:extractant ratio to extract bulk solution and exchangeable ions from the soil.

Although these three extractant systems constitute only a small part of the total number used throughout the world, they do represent the principal types used in the

southeastern portion of the U. S. Several methods have been proposed for the determination of individual cations in soil extracts (184, 176, 249, 82, 78). These methods are often determined by the type of extractant and the concentration of ions in the solution (68).

For those extractant systems previously described, atomic absorption spectrophotometry is well adapted. This method is quick, accurate and convenient for the determination of K, Ca, Mg, Mn, Fe, An, Cu, Na and Al in soil extracts.

Phosphorous (P) determinations have received considerable attention through the years. Although several complex analytical systems have been proposed, no one method has been found universal.

Primary interest in soil P has centered around the "available" fraction. "Available" P refers to the amount of the element that can be absorbed from the soil by plants. The underlying principle of these methods is to extract a soil sample with a solution designed to dissolve the fractions of P that are available to plant roots. The extract is then analyzed for soluble P and the results correlated with actual P taken up by the plant (164, 8).

Although several types of extractants have been proposed (159, 46, 72, 75, 57, 168), dilute, acid-fluoride containing solutions have had broadest implications (33). The  $F^-$  ion has the ability to complex  $Al^{+++}$  and  $Fe^{+++}$  ions in acid solution with the consequent release of P held in the soil by the trivalent forms of these ions (231).

Various modifications of this extractant have been developed for calcareous soils. In the extraction, a higher concentration of acid is used (Bray No. 2) and adjustments made in extraction time (28). Further modifications have been developed for specific areas and situations (64).

Once in solution, P can be estimated volumetrically, gravimetrically or colorimetrically. Of the most commonly employed methods of determining P in solution, the latter is most commonly used. Two principal colorimetric procedures exist, each of which has advantages over the other under certain circumstances. One depends upon the combination of P with the molybdate ion and the other upon its reaction with vanadomolybdate (121, 204).

Of the many reducing agents used for the colorimetric determination of P, ascorbic acid has become favored. This is attributed to the fact that the blue color of the reduced complex remains stable for a relatively long period of time, allowing reduced solutions to be kept overnight if necessary (210). One of the disadvantages of this method is that during the long period of time required for color development, some organic P may be hydrolyzed. However, modifications have been developed which decrease the time needed and allow for the determination to be conducted in aqueous soil extracts (230, 180).

The vanadomolybdate method is also commonly used for the determination of P in soil extracts. In this method a yellow color is developed by the addition of excess molybdate ions to an acid solution of vanadate and orthophosphate



(130). The color is more stable than molybdenum blue and the method is subject to less interference. Best results are obtained under constant temperature and after approximately thirty minutes for color development (108).

The Kjeldahl (118) method is the most commonly used for the determination of total nitrogen (N). This procedure converts organic N into ammonia by boiling with sulfuric acid. This ammonia is then liberated from its sulphate by distillation with alkali and estimated. Although this procedure is generally considered rather simple, it is subject to several variables which may lead to inaccurate values.

The original process of simple digestion with acid has been modified to include the addition of potassium sulphate to raise the boiling point of the acid (222). The consequent effects on the temperature of digestion have been studied in great detail. It has been shown that during the digestion procedure some acid is lost by: volatilization, reaction with soil minerals and by the oxidation of organic matter. Such acid loss causes an increased salt concentration and, hence, a higher boiling point (173, 76, 188). These factors must be controlled to avoid N loss from the system.

Several catalysts have been recommended to hasten the Kjeldahl reaction. Those most commonly used are copper sulphate, mercury oxide and selenium. The interaction between these various catalysts and the time of digestion have been studied to determine their effect on N values (124, 166). Although this is somewhat dependent on soil

type, there is evidence which supports the use of longer digestion procedures in most cases (148, 10). It has further been shown that the clearing of the digestion mixture is not a criterion of complete digestion (200).

The routine Kjeldahl method does not measure certain forms of N. The failure to estimate nitrate-N and nitrite-N may be corrected by modifications in which salicylic acid and sodium thiosulphate are added to the sulfuric acid used for digestion (187). In most soils the nitrate and nitrite content is negligible, and little harm is done by neglecting a pre-treatment. However, if nitrates are likely to be present in significant quantities, they should be included in a total N analysis or determined separately (36).

Modifications have also been proposed for the inclusion of free ammonium in the total N analysis (93). It has been demonstrated that considerable differences exist between the sum of the N fractions and Kjeldahl N when such modifications are not utilized. Pre-treated soils, however, have been shown to yield total Kjeldahl N contents very close to the sum of N fractions (138).

Adaptations of the Kjeldahl procedure have been developed which reduce sample size and liberated gases. These micro kjeldahl methods are quicker, neater and consume far smaller quantities of chemicals (236). Micro procedures have been recommended for soil samples which vary from 1 g for peats to 20 g for sands. Digestion of these materials generally requires 20 g of catalyst and 35 cc of sulfuric acid (195).

Ammonium is subject to microbial changes and determination should quickly follow the collection of soil samples. These samples may be extracted moist, or dried rapidly at 50°C (63). Since most ammonium is held in exchangeable form by soils, it may be displaced by some other exchangeable cation (91, 30). Although several extraction procedures have been proposed (2), equilibrium extractions have been shown to be the most satisfactory and rapid (31).

Three methods are in current use for determining the ammonium content of a solution. The first depends upon alkaline steam distillation, in which liberated ammonia is absorbed in acid and titrated (200); the second depends upon the reaction of ammonia with Nessler's reagent to give a colored solution; and the third is a micro-diffusion method (171).

Nitrate nitrogen is also subject to microbial changes and should be determined immediately after sampling (108). The same reagents used for ammonium extractions are used for extracting nitrate. Generally, ammonium and nitrate determinations are conducted in the same extract and, with some methods, in the same aliquot of extract. Nitrates may be determined by either reduction to ammonia, which is then liberated by alkaline steam distillation and titrated, or determined colorimetrically after reduction to nitrite or ammonia (94, 185). The most important of these colorimetric determinations is the nitrophenoldisulfonic yellow color method (249).

Carbon occurs in soils in the elemental, inorganic and

organic forms. The principle of total carbon analysis is to convert the element into  $\text{CO}_2$  which is then determined gravimetrically or volumetrically. Oxidation is achieved by combustion, either wet or dry, and all forms of carbon can be included. Although the total carbon content of a soil is seldom determined, when necessary the analysis is best made by dry combustion (185). This method requires a furnace fitted with some means of collecting the  $\text{CO}_2$  evolved. A schematic diagram of a combustion train for the determination of total carbon is presented in Figure 1.

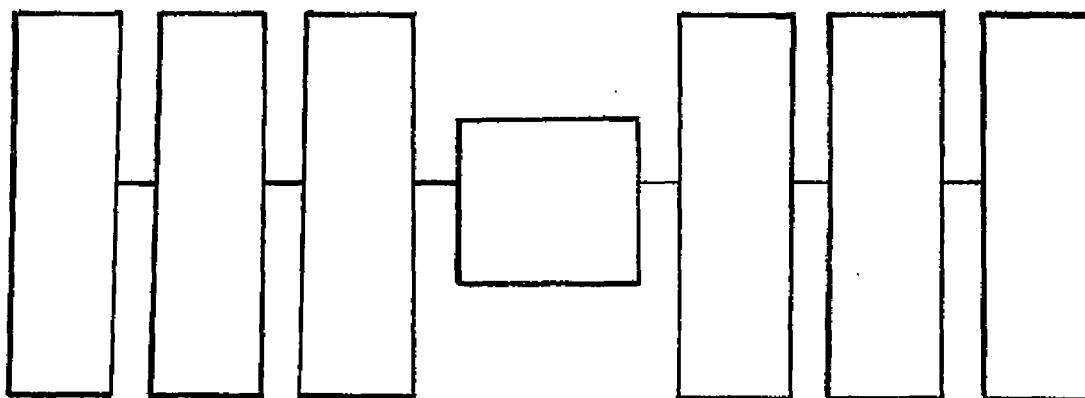


Figure 1. Schematic diagram of a standard Fisher  $\text{CO}_2$  combustion train.

Combustion is carried out in a stream of pure oxygen (a) which is scrubbed with sulfuric acid (b) and passed through ascarite and magnesium perchlorate to remove any  $\text{CO}_2$  or hydrocarbons. Sulfur dioxide is trapped by manganese dioxide, water vapor is removed by anhydrous magnesium perchlorate and carbon monoxide is oxidized over platinum metal. This oxidized carbon monoxide is collected as  $\text{CO}_2$  in an absorption weighing tube containing a layer of

magnesium perchlorate over ascarite. Samples are oxidized at a maximum of 1000°C for one minute (236, 108).

The Nutrition of *Euphorbia pulcherrima* Willd:

Qualitative deficiency symptoms of *Euphorbia pulcherrima* Willd. have been reported by Widmer (245) and by Laurie and Wagner (123). These studies have shown that, although N, P, K, Ca, Mg and B deficiencies can be induced, symptoms do not always develop at the same time or in the same manner in different plants of the same cultivar. It has also been shown that those cultivars or individual plants making a more rapid growth usually develop symptoms first.

In a more quantitative approach, Struckmeyer (220) studied the leaf anatomy of poinsettia plants grown with some inadequate supplies of nutrient elements. Her findings indicated that the abnormal leaf anatomy associated with a particular deficiency could be used in identifying the inadequate element. Necrotic lesions, hypertrophy and hyperplasia, chloroplast disintegration, accumulation of cellular inclusions, and starch accumulations were all found to be characteristics which accompanied various mineral deficiencies.

Much of the work on poinsettia nutrition has been directed towards fertility as related to commercial production. Shanks and Link (208) studied stock plant nutrition in relation to the production, rooting and growth of cuttings. Their findings showed that the interactions between N, P and K significantly influenced the growth potential of

cuttings from all treated plants. In a later study (128) they further demonstrated a relationship between P and Mg, which under the conditions of their experiment, varied with cultivar.

Foliar analysis has become an important method of assessing the nutritional status of plants. Boodley (19) has established a sampling and analytical technique which has generally been accepted as the most valid estimation of the nutrient content of poinsettias. Based on data collected from this procedure, a tentative key has been developed for the interpretation of foliar analysis values (67) (Table IV). Although the overall range for these elements is quite wide, more precise values will evolve as reasearchers increase their use of foliar analysis (66).

A number of studies have examined the effect of N form on the growth and quality of finished poinsettia plants (117, 21, 125). It has been shown that excessive quantities of ammonium or urea may cause poor root development, yellowing, leaf drop and stunting. Therefore, it has been recommended that no more than one-half of the N supplied poinsettias be in the form of ammonium (20). Nitrate nitrogen in the form of potassium nitrate, calcium nitrate and ammonium nitrate are generally considered the most suitable sources for the supplemental application of N.

Poinsettias have long been thought to require relatively high K fertilization (66, 87). However, Shanks and Link (207) have postulated that with an adequate supply of nutrients, particularly N, the use of high K fertilization

may be unnecessary. Furthermore, they noted that Mg deficiencies may be overcome by the dual application of N and K. Stuart and Rocke (221) also found that K deficiencies did not limit growth as much as did deficiencies of N, P or Ca. This study also demonstrated interdependencies between K, Ca and Mg which had a significant effect on growth.

Table IV. Foliar analysis interpretation key for Euphorbia pulcherrima Willd.<sup>1</sup>

Element	Critical level	Normal range	Toxic level
Nitrogen (%)	3.0	4.0 - 6.0	37.3
Phosphorus (%)	0.2	0.3 - 0.7	30.7
Potassium (%)	1.0	1.5 - 3.5	4.0
Calcium (%)	0.5	0.7 - 2.0	-
Magnesium (%)	0.2	0.4 - 1.0	-
Sodium (%)	-	0.0 - 0.4	0.5
Chloride (%)	-	0.0 - 0.7	1.0
Copper (ppm)	1.0	2.0 - 10	-
Zinc (ppm)	15	25 - 60	-
Manganese (ppm)	40	80 - 300	650
Iron (ppm)	50	100 - 300	-
Boron (ppm)	20	30 - 300	700
Molybdenum (ppm)	0.5	1 - 5	-

<sup>1</sup>Based on oven-dry tissue.

Relatively little work has been conducted on the micro-nutrient requirements of poinsettias. Kofranek, Lunt and Kohl (120) studied the effect of B uptake and found that B levels of 4.8 ppm and above caused interveinal chlorosis and marginal leaf scorch, followed by leaf abscission. Similar symptoms have also been related to toxic levels of Cu, Zn and Mn (125).

Perhaps Mo has received more attention than any of the other micronutrients in poinsettia nutrition. Although Mo toxicity may be a problem in certain areas, it is generally the deficient condition which most affects poinsettias. Symptoms of Mo deficiency have been described as the yellowing of mature leaves, sometimes progressing to a leaf edge burn. Because Mo is required for nitrate reduction, a test for  $\text{NO}_3$  in leaf tissue is an indirect means of determining Mo status. Severe Mo deficiencies have generally been associated with  $\text{NO}_3$  values varying from 6,000 to 14,000 ppm (181).

Another factor to be considered in poinsettia nutrition is the pH of the growing medium. The solubility of many nutrient elements is affected by pH. Generally Cu, Zn, Mn, Fe and Al become less soluble as pH rises. However, too low a pH may result in micronutrient toxicity and low Ca-Mg supplies. Molybdenum becomes more soluble at high pH, although micronutrient deficiencies and Na toxicity may result if pH becomes too high (71).

Poinsettia growth has been shown to be most tolerant to pH levels ranging from 5.5 to 6.5. However, the use of



soilless growing media and advanced fertilization methods have reduced some of the effects of pH on nutrient availability (6).

Many methods of supplying fertilizer to plants have been studied. These include mist fertilization (158), liquid fertilization (77, 116), uses of slow-release fertilizers (135), and the use of dry (granular) fertilizers (206). Although no particular method may be conclusively regarded as the best program, liquid fertilization is the most popular. Ecke (67) has recommended a liquid fertilization program suitable for a wide range of poinsettia cultivars (Table V). However, Criley and Parvin (56) have demonstrated the advantages of a combination of liquid and slow-release fertilization in maintaining optimum nutrient levels.

Table V. Liquid fertilization programs for Euphorbia pulcherrima Willd.

Constant liquid feed	
Source	Amount/10 cu m water applied
Ammonium Nitrate	3.6 Kg
Calcium Nitrate	6.0 Kg
Potassium Nitrate	3.6 Kg
75% Phosphoric Acid	1250.30 cc
Molybdenum stock solution <sup>1</sup>	40.40 cc
Intermittent liquid feed	
Source	Amount/10 cu m water applied
Ammonium nitrate	6.6 Kg
Calcium nitrate	12.0 Kg
Potassium nitrate	6.6 Kg
75% Phosphoric acid	2500.60 cc
Molybdenum stock solution <sup>1</sup>	80.80 cc

<sup>1</sup>Molybdenum stock solution: 190.15 g ammonium molybdate/liter of water

## MATERIALS AND METHODS

The bark used in this study represents a composite of pine species but may be considered typical of that used in the preparation of growing media throughout the Southern Gulf States.

### Determination of Particle Size Distribution and Bulk Density of Southern Pine Bark:

A pre-screened sample of bark (1/4 in mesh) was placed in a previously tared 1000 cc sampler. The base of the sampler was tapped on the counter top until settling was no longer detectable. The sampler and its contents were placed, overnight, in a drying oven at 100°C. The weight of the dried bark was then determined and bulk density calculated:

$$BD \text{ (g/cc)} = \text{Oven-dry wt}/1000 \text{ cc}$$

The contents of the sampler were screened through NBS sieve nos. 4, 10 and 20, and the weight of each fraction recorded. The bulk density of each fraction was then calculated:

$$BD \text{ (g/cc)} \text{ of each fraction} = \frac{\text{Oven-dry wt of each fraction}}{1000 \text{ cc}}$$

### Determination of the Specific Gravity of Southern Pine Bark:

A clean, dry, 100 ml volumetric flask was weighed in air. A 5 g sample (air dry) of screened bark (NBS sieve

no. 4) was placed in the flask and reweighed (including stopper) (a duplicate sample was run for moisture).

The flask was filled approximately one-half full with distilled water and boiled gently to remove entrapped air. The flask was cooled to room temperature and brought to volume with boiled, cooled, distilled water at room temperature. The flask was stoppered, weighed and the temperature of its contents determined.

The bark sample was removed from the flask and the weight of the flask, filled with boiled, cooled, distilled water at room temperature, was recorded (with stopper). Specific gravity was then calculated:

$$\text{Specific Gravity} = \frac{d_w (W_s - W_a)}{(W_s - W_a) - (W_{sw} - W_w)}$$

where:  $d_w$  = density of water (g/cc) at temperature observed

$W_s$  = weight of flask plus bark corrected to oven-dry condition

$W_a$  = weight of flask filled with air

$W_{sw}$  = weight of flask filled with bark and water

$W_w$  = weight of flask filled with water at temperature observed

#### Determination of the Container Capacity of Southern Pine Bark:

Container capacity was determined in containers (17.2 cm deep, 10.5 cm dia.) with perforated bases, and filled with a disk of qualitative filter paper (Whatman no. 42). Screened bark (NBS sieve no. 4) was placed in the containers and tapped on the counter top until settling was no longer

detectable.

The filled containers were placed into 2 gal plastic buckets fitted with a wire mesh base to permit water movement. The buckets were filled with water to a level 1 cm below the surface of the bark and then fitted with lids to prevent evaporation. The entire system was allowed to equilibrate for 24 hours. The water in the buckets was drained out and gravitational water allowed to drain from the bark for 5 minutes. This procedure was repeated 3 times.

The weight of the container and its contents, following the last drainage cycle, was used in calculating the weight of water held by the bark. The contents of the container was removed, weighed and oven dried at 100°C until it reached a constant weight. The wet and oven-dry weights were used to calculate the weight of moisture held. The weight of moisture per unit weight of oven-dry bark and the bulk density were then used to calculate volume percentages.

#### Determination of the Percolation Rate of Southern Pine Bark:

Percolation rate was determined in containers (17.2 cm deep, 10.5 cm dia) fitted with a wire gauze base (coarse enough as not to impede water movement) and a series of perforations 1.2 cm from the top of the container. Screened bark (NBS sieve no. 4) was placed in the container and tapped on the counter top until settling was no longer detectable. This procedure was repeated until the container was filled to the 15 cm mark.

The filled container was placed in a 2 gal bucket which

was then filled with water to a level 1 cm below the surface of the bark. This unit was fitted with a lid and the entire system allowed to equilibrate for 24 hours. The water in the bucket was drained out and gravitational water allowed to drain from the bark for 5 minutes. This procedure was repeated 3 times.

The container was removed from the bucket and the volume of bark adjusted for shrinking and swelling to the 15 cm mark. Water was moved through the container for 15 minutes using an over flow system to maintain a constant head of 1 cm. Drain water was collected in a 1000 ml graduated cylinder fitted with an aluminum foil drip shield. The rate of water percolation was then calculated:

$$\text{Percolation rate (c/15 min)} = \frac{Ql}{TAh}$$

where: Q = volume of water

T = time

A = cross-sectional area

l = length

h = loss in hydraulic head through l

#### Determination of an Adjusted Particle Size Distribution and Bulk Density for Analytical Samples of Southern Pine Bark:

A sample of pre-screened bark (1/4 in mesh) was placed in a previously tared, 50 cc vial. The base of the vial was then tapped on the counter top until settling was no longer detectable. This procedure was continued until the vial contained 45 cc's of sample material. The vial and its contents were placed overnight in a drying oven at 100°C.

The weight of the dried bark was then determined and bulk density calculated:

$$\text{BD (g/cc)} = \text{Oven-dry wt of bark} / 45 \text{ cc}$$

The contents of the vial was screened through NBS sieve nos. 4, 10 and 20 and the weight of each fraction recorded. The bulk density of each fraction was then calculated:

$$\text{BD of Screened Fraction (g/cc)} = \frac{\text{Oven-dry wt of fraction}}{45 \text{ cc}}$$

This procedure was then repeated using 45 cc samples of bark pre-screened through NBS sieve no. 4.

#### Determination of Total Nitrogen in Southern Pine Bark:

Total N was determined with a standard Kjeldahl distillation apparatus using the following procedure: A 5 g sample of screened bark (NBS sieve no. 4), air dry, was placed in a 500 ml Kjeldahl flask (additional blanks were run as a reagent check). Approximately 10 ml of distilled water were used to wash all sample material to the bottom of the flask. Twenty-five ml of concentrated sulfuric acid and 15 g of Kjeldahl salt mixture ( $\text{K}_2\text{SO}_4$ ,  $\text{FeSO}_4$ ,  $\text{CuSO}_4$ , 10:1:1/2 v/v/v) were added to the flask.

The flask, with its contents, was placed on the digestion rack and heated for about 2 hours (until the solution became clear to bluish in color, indicating completed digestion). The flask was allowed to cool before the addition of 200 ml of water.

One hundred ml of a 2% boric acid solution were placed

in a 500 ml Erlenmeyer flask, which was then placed at the end of the delivery tube on the distilling apparatus. One hundred ml of a 45% NaOH solution were added to the contents of the Kjeldahl flask along with about 6 Zn pellets. This flask was then immediately stoppered and its contents mixed with a swirling motion.

Approximately 150 ml of water (containing N as ammonia in solution) were distilled over to the Erlenmeyer flask. Two drops of methyl red indicator were added to the distillate and then titrated with standard 0.1000 N HCl. The amount of acid used was recorded with appropriate corrections made for the blank. Percent total N was then calculated:

$$\% \text{ Total N} = \frac{\text{ml } 0.1000 \text{ N HCl} \times 0.0014 \times 100}{5 \text{ g sample}}$$

#### Determination of Total Phosphorus in Southern Pine Bark:

A 5 g sample of screened bark (NBS sieve no. 4), oven-dry (60°C), was ashed in a muffle furnace at 500°C for 4 hours. The ash was taken up in 10 ml of 8 N HCl, diluted to 100 ml with distilled water and filtered through Whatman no. 42 filter paper.

A 10 ml aliquot of filtrate was placed in a 50 ml volumetric flask to which was added 10 ml of vanadomolybdate reagent (prepared as follows: Solution A, 25 g ammonium molybdate in 400 ml of water; Solution B, 1.25 g of ammonium metavanadate in 300 ml boiling water, cooled, 250 ml concentrated HNO<sub>3</sub> added. Solutions A and B added together and diluted to one liter). The solution was brought to volume



with distilled water.

Percent transmission was read on a spectrophotometer at 400-490 mu against a reagent blank. These readings were plotted on a standard curve (prepared from  $\text{KH}_2\text{PO}_4$  and 7.0 N  $\text{H}_2\text{SO}_4$  standards) calibrated in parts per million. Percent P was then calculated:

$$\% \text{ P} = (\text{ppm P}) \times 100/1,000,000$$

Determination of Total Potassium, Calcium, Magnesium, Iron, Zinc and Manganese in Southern Pine Bark:

A 5 g sample of screened (NBS sieve no. 4), oven-dry (60°C) bark was ashed in a muffle furnace at 400°C for 4 hours. The ash was taken up in 10 ml of 8.0 N HCl, diluted to 100 ml with distilled water, and filtered through Whatman no. 42 filter paper.

Appropriate dilutions of the filtrate were then made and the concentrations of K, Ca, Mg, Fe, Zn and Mn determined by means of atomic absorption spectrophotometry.

Determination of Available Phosphorus, Potassium, Calcium, and Magnesium in Southern Pine Bark:

Twenty-five cc samples of screened (NBS sieve no. 4) bark were placed in 125 ml conical flasks and extracted for 1, 2, 3, 4, 5, 6, 12 and 24 hours using three extractants (0.025 N HOAc, 1.0 N  $\text{NH}_4\text{OAc}$  pH 7.0 and equal parts of 0.025 N HCl and 0.05 N  $\text{H}_2\text{SO}_4$ ) in a 1:4 v/v substrate:extractant ratio. These solutions were filtered through Whatman no. 42 filter paper. The concentrations of K, Ca and Mg were then determined by means of atomic absorption

spectrophotometry. Concentrations of P were determined in aliquots of the same extract using the vanadomolybdophosphic yellow method.

#### Determination of Carbon as CO<sub>2</sub> in Southern Pine Bark:

Carbon was determined with a standard Fisher CO<sub>2</sub> combustion train using the following procedure: An alundum boat was lined, half full, with granular alundum and covered with 0.5 g powdered electrolytic iron. A 0.2727 g sample of screened (NBS sieve no. 4), ground bark (air dry) was mixed with 0.5 g of a 50:50 mixture of granular tin and electrolytic iron power combustion accelerator. This mixture was quantitatively transferred to the prepared boat and covered with an additional 0.5 g of electrolytic iron powder. The boat, with sample, was placed in the combustion furnace at 1000°C for approximately 2 minutes.

The weight of the previously tared ascarite bulb was recorded and the weight of CO<sub>2</sub> determined. Percent carbon was then calculated:

$$\% \text{ Carbon} = \frac{\text{weight of CO}_2 \text{ (mg)}}{10}$$

#### Determination of Acid Insoluble Lignin in Southern Pine Bark:

A 2 g sample of screened bark (NBS sieve no. 4), air dry, was placed in a 500 ml beaker. One hundred ml of acid detergent solution (20 g cetyl trimethylammonium bromide in 1 liter of 1.0 N H<sub>2</sub>SO<sub>4</sub>) and 2 ml decahydronapthalene were then added. The solution was placed on a refluxing apparatus and heated to boiling. Refluxing was continued

for 60 minutes from the onset of boiling.

The solution was filtered using a previously tared, fritted glass crucible, with light suction. Samples were washed with hot water and acetone until color could no longer be removed. The crucible and its contents were dried at 100°C for approximately 8 hours and cooled in a desiccator over phosphorous pentoxide and then weighed.

The crucible was placed in a 50 ml beaker, for support, and its contents covered with cooled (15°C) 72% H<sub>2</sub>SO<sub>4</sub>, stirring and replacing acid hourly. After 3 hours, as much acid as possible was filtered off with suction and the sample washed with hot water. The crucible and its contents were dried at 100°C for approximately 8 hours and cooled in a desiccator over phosphorus pentoxide and weighed.

The contents of the crucible were ignited in a muffle furnace at 500°C for 2 hours and placed, while still hot, in a desiccator, cooled and weighed. Percent acid detergent lignin was then calculated:

$$\% \text{ Acid detergent lignin} = \frac{(L \times 100)}{S}$$

where: L = loss of weight upon ignition after  
72% H<sub>2</sub>SO<sub>4</sub> treatment

S = oven-dry sample weight (obtained by  
conducting a separate dry matter de-  
termination and multiplying % dry  
matter by the air-dry weight)

#### Determination of the Cation Exchange Capacity of Southern Pine Bark:

Two index extractants (1.0 N BaCl<sub>2</sub> pH 8.0 and 1.0 N NH<sub>4</sub>OAc pH 7.0), two substrate:extractant ratios (1:2 v/v

and 1:4 v/v), and two extraction times (2 hours and 24 hours) were placed in a factorial arrangement of treatments using a completely random design, with 6 observations per treatment combination. Cation exchange capacity was determined using the following procedure:

Twenty-five cc samples of screened bark (NBS sieve no. 4) were prepared using the appropriate treatment combination in a 125 ml conical flask. The contents of the flask were transferred to a Buchner funnel and filtered through Whatman no. 42 filter paper using light suction. The flask and sample were washed with 100 ml of 95% ethanol using a squirt bottle.

The Buchner funnel containing the sample was transferred to a new receiver and extracted with three 30 ml aliquots of displacement solution (10%  $\text{NaCl}_2$  for the  $\text{BaCl}_2$  extraction and 10%  $\text{KCl}$  for the  $\text{NH}_4\text{OAc}$  extraction). The contents of the receiver were transferred to a 100 ml volumetric flask, washed and made to volume with the appropriate displacement solution. The concentration of Ba and  $\text{NH}_4$  ions were then determined and exchange capacity calculated.

#### Determination of the pH of Southern Pine Bark:

Two solutions (water and 0.01 N  $\text{CaCl}_2$ ), two substrate: solution ratios (1:2 and 1:4 v/v), and two equilibration times (30 min and 24 hrs) were placed in a factorial arrangement of treatments using a completely random design with 6 observations per treatment combination. The pH of 25 cc samples of screened bark (NBS sieve no. 4), prepared using

each treatment combination, was then determined by means of a calibrated glass electrode pH meter.

Determination of Exchangeable Hydrogen and Aluminum and Total Exchangeable Acidity of Southern Pine Bark:

A 25 cc sample of screened bark (NBS sieve no. 4), air dry, was placed in a Buchner funnel fitted with filter paper (Whatman no. 42) and extracted with 100 ml of 1.0 N KCl using light suction. Five drops of phenolphthalein indicator (0.1 g powder in 100 ml ethanol) were added to the filtrate and titrated with 0.1 N NaOH.

One drop of indicator and 10 ml of NaF solution (40 g NaF in 1 liter water) were added to the filtrate and this solution titrated with 0.1 N HCl. Exchangeable Al, H and total exchangeable acidity were then calculated:

Total exchangeable acidity (meq/100cc) = (ml NaOH X 4) X N HCl

Exchangeable Al (meq/100 cc) = (ml HCl X 4) X N HCl

Exchangeable H (meq/100 cc) = (total exchangeable acidity - exchangeable Al)

The Use of Plant Uptake to Determine Optimum Values for the Analysis of Pine Bark Growing Medium:

Rooted cuttings of 'Annette Hegg Dark Red'<sup>1</sup> were panned in standard 15.24 cm clay pots, using an amended pine bark growing medium (Table VI). These plants were placed on artificially long days for a period of two weeks to establish vegetative growth. At the start of short days, all

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<sup>1</sup>Sincere appreciation is expressed to Paul Ecke Poinsettias, Encinitas, CA, for donation of plant materials.

Table VI. Composition of amended pine bark growing medium.

Source	Amount/cu m of milled pine bark
Dolomitic lime	18.67 kg
Superphosphate	12.45 kg
FTE <sup>1</sup>	85.96 g

<sup>1</sup>Fritted trace elements.

Table VII. Composition of recommended (1.0 X) fertilizer solution.

Source	Amount/10 cu m water applied
Ammonium nitrate (33% N)	3.6 kg
Calcium nitrate (15% N,	6.0 kg
Potassium nitrate (14% N, 44% K)	3.6 kg
Phosphoric acid (75% P)	1250.30 cc
Molybdenum stock solution <sup>1</sup>	44.40 cc

<sup>1</sup>Molybdenum stock solution: 190.15 g ammonium molybdate/liter of water.

Table VIII. Fertilizer solutions applied to Euphorbia pulcherrima Willd. cv 'Annette Hegg Dark Red.'

Treatment	Element (ppm)		
1. (0.25 X)	64.75	10.75	33.00
2. (0.5 X)	129.50	21.50	66.00
3. (1.0 X)	259.00	43.00	132.00
4. (1.5 X)	385.00	64.50	198.00

plants were pinched to 4 nodes per plant above medium level.

Four treatment fertility regimes were derived from those rates recommended by Ecke (67) (Table VII). These regimes were based on the calculated amount of each element which the plant could utilize during the course of the experiment (Table VIII). All treatment regimes were initiated at planting by means of a constant-liquid feed program (approximately 650 ml/watering) using a trickle irrigation system.

Two sampling dates were selected to determine possible shifts in nutrient utilization during the growing season. Sampling date one (four weeks after the start of short days) was used as an estimate of demand during active growth. Sampling date two (at the initiation of bract color) was used as an estimate of demand during reduced growth. On appropriate dates, plants were sampled for foliar analyses following the procedure described by Boodley (19). Foliar samples were placed in a drying room at 75°F for 2 days. Dried samples were then thoroughly ground using a mortar and pestle and placed in labeled, plastic sample bags for subsequent analysis:

A 0.5 g sample of dried tissue was placed in a porcelain crucible and ashed in a muffle furnace for 4 hours at 550°C. The ash was taken up in 10 ml of 8.0 N HCl and the sample filtered through Whatman no. 42 filter paper (washing with hot, distilled water) into a 50 ml volumetric flask. The sample was brought to volume with distilled water, and the concentrations of K, Ca, Mg, Fe, Zn and Mn

determined by means of atomic absorption spectrophotometry. Total P was determined from a 10 ml aliquot of filtrate using the vanadomolybdophosphic yellow procedure previously described.

Total N was determined with a micro-Kjeldahl apparatus using the following procedure: A 0.10 g sample of tissue was folded into a 2" X 2" piece of lab tissue and placed in a Kjeldahl flask (a blank with only tissue was run simultaneously). To the flask was added 2 g of digestion salt ( $K_2SO_4$ ,  $FeSO_4$  and  $CuSO_4$ , 10:1:1/2 v/v/v) and 4 ml of concentrated  $H_2SO_4$ . Samples were digested for 1 hour, after they became clear, and cooled for 20 minutes prior to the addition of 20 ml of de-ionized water.

Ten ml of 0.1 N  $H_2SO_4$  were added to a 50 ml Erlenmeyer flask which was then placed on the end of the outlet tube on the distillation apparatus.

The Kjeldahl flask and its contents were attached to the still and 20 ml of 50% NaOH were added through the reservoir. Approximately 30 ml of distillate were distilled over to the Erlenmeyer flask and 2 drops of mixed indicator (0.5 g bromcresol green + 0.1 g methyl red in 100 ml of 95% ethanol) added. The distillate was then titrated with 0.10 N NaOH and the percent N calculated:

$$\% N = (B - T) \times \underline{N} \times (1.4) / \text{Sample wt}$$

where: B = ml NaOH for blank

T = ml NaOH for sample

N = normality of NaOH



Substrate analysis was conducted on 12 samples from each treatment combination. Standardized 25 cc samples of bark were extracted for 24 hours with 0.025 N HOAc, 1.0 N NH<sub>4</sub>OAc pH 7.0, and equal parts of 0.05 N HCl and 0.025 N H<sub>2</sub>SO<sub>4</sub> using a 1:4 (v/v) substrate:extractant ratio. These solutions were filtered and the concentrations of P, K, Ca and Mg determined in the filtrate.

Treatments were arranged in a split plot design with fertility regimes on the main plot and sampling date on the split plot. Each treatment combination was replicated 6 times with 2 sub-samples per replication.

## RESULTS AND DISCUSSION

### The Physical Properties of Southern Pine Bark:

A complex relationship exists between the physical and chemical properties of soilless growing media. This relationship is further confounded by the use of frequent irrigation-fertilization regimes for the production of most containerized floricultural crops. Although the primary objective of this study was to examine the chemical properties of pine bark medium, it was essential to first characterize its basic physical properties. The results from these determination are presented in Table IX.

Particle size distribution indicates the percentage of separates, on a weight basis, within designated particle size ranges. The ratio of these particles to one another determines many of the aeration, drainage, and water-holding characteristics of a growing medium. Therefore, a knowledge of the particle size distribution of a growing medium may be used as a background to interpret many of its physical and chemical properties.

The particles retained on the no. 4 sieve represented the smallest percentage of total separates. However, these coarse particles most affect the size of pores and the overall matrix of the medium. The particles retained on

Table IX. Physical properties of southern pine bark.<sup>1</sup>

Particle distribution (%/wt)

NBS sieve size				Receiver pan	Bulk density (g/cc)	Specific gravity (g/cc)	Percent pore space (%/vol)	Container capacity (%/vol)	Percolation rate (cm/15 min)
4	10	20							
Openings (mm)									
4.76	2.00	0.84	<0.84						
16.11 <sup>1</sup>	31.00	25.06	27.75	0.274	0.762	64.041	36.346	84	
0.286 <sup>2</sup>	0.06	0.06	0.17	0.03	0.04	0.45	0.30	0.39	
1.42 <sup>3</sup>	0.30	0.30	0.85	0.15	0.20	2.26	1.50	1.95	

<sup>1</sup>Means of 25 samples.

<sup>2</sup>Standard error.

<sup>3</sup>Standard deviation.

the no. 10 and 20 sieves and the receiving pan represented the largest percentage of total separates. These particles are primarily responsible for the determination of the number of pores, and they also constitute the most reactive particles of the medium.

The relationship between pore size and number influences many of the physical and chemical properties of bark medium. Therefore, it is essential to maintain an adequate distribution of particles.

Bulk density is an important characteristic which influences both the physical and chemical properties of a growing medium. Although bulk density must be low enough to promote good aeration and drainage, it must also be high enough to provide adequate anchorage for the plant. This is particularly true for the production of containerized floricultural crops.

Bulk density is largely determined by compaction. Excessive pressure applied during potting may substantially increase bulk density and reduce aeration and drainage. Compaction may also occur as the result of improper irrigation techniques. Water applied at high pressure will cause the surface layer of media to compact, increasing the bulk density, and reducing aeration and drainage.

Based on the results from the determination of the bulk density, it may be concluded that the weight per unit volume is sufficient for both adequate anchorage and those physical properties required for plant growth. Further, the relatively small amount of variation between bulk density

values for 25 samples indicates the uniformity of the growing medium.

Bulk density is also affected by the average density of bark particles, or specific gravity. Generally, as specific gravity increases, bulk density increases.

Of primary concern in the determination of specific gravity of any organic material is the problem of "floating." The tendency of bark particles to rise to the surface of the filled pycnometer creates difficulty in obtaining a measurement of volume. However, with careful handling, this problem may be overcome.

Little work has been conducted on the specific gravity of bark medium. Therefore, it is difficult to interpret the results from Table IX. However, these values do agree with the limited data previously reported (140).

The total percent pore space of a growing medium may be calculated from the relationship between bulk density and specific gravity. Given these two values, the percent of the volume occupied by solids is:

$$\% \text{ Solid} = \frac{\text{bulk density}}{\text{specific gravity}} \times 100$$

Therefore, the percent of the volume occupied by pore space is:

$$\% \text{ Pore space} = 100 - \% \text{ solids}$$

Pore space should occupy 1/3 to 1/2 of the total volume of a growing medium in order to provide proper aeration and

drainage properties. Therefore, it may be concluded that the porosity of the bark medium is adequate for plant growth.

The moisture content of a growing medium continuously changes according to the water potentials that develop. The concept of container capacity assumes the development of a constant state in which surplus water has drained from the medium in the absence of evaporation. Therefore, the container capacity of a growing medium represents the upper limit of available water. Although this is an important measurement of the moisture holding capacity of a growing medium it exists only in theory.

The container capacity of the bark medium has been shown to approach that of peatmoss, and other soilless constituents. Therefore, it may be concluded that the moisture holding capacity of this medium is suitable for use in the production of containerized floricultural crops.

The percolation rate of a growing medium is an important property in relationship to the rate of irrigation-fertilization. Because most containerized floricultural crops are grown at relatively high rates of fertility, additional quantities of water in excess of container capacity are required to regulate the levels of soluble salts. Therefore, the percolation rate indicates the rate at which irrigation-fertilization regimes may be applied.

Growing media with relatively low percolation rates require lower rates of application. These media are often suitable only for drip or trickle irrigation. However, the percolation rate of the bark medium was not found to indicate

such restrictions.

Although these physical properties were determined independently, it is their interaction which determines the aeration, drainage and moisture holding characteristics of pine bark growing medium. Since frequent irrigation-fertilization regimes are used to maintain most containerized floricultural crops, the relationship between the physical and chemical properties of the growing medium is apparent. Therefore, meaningful interpretations of analytical values may be made only when this relationship is clearly defined.

The accuracy and precision of any analytical determination is largely dependent upon the homogeneity of the material being tested. Therefore, when considering the complexity of an analytical system such as soilless growing media, it is essential to recognize potential sources of variability. Once these sources have been identified, appropriate measures must be taken to account for these variables without interfering with the objectives of the analysis. This, to a great extent, is contingent upon the method of sampling and sample preparation. Although many of the principles of soil sampling apply to bark media, certain adaptations are required.

The sampling volume for most containerized floricultural crops is relatively small. Therefore, the potential for sampling error is reduced. However, this is dependent upon the random selection of an adequate number of samples which represent the entire sampling volume.

The method of selecting random samples is dependent upon the status of the sampling volume itself. Samples taken from bulk media, for pre-plant analysis, must be representative of the entire mass of growing medium. Therefore, a device such as a sampling tube should be used to collect samples from various locations within the sampling volume. Samples taken from the containers of actively growing plants, for post-plant analysis, must also be representative of the entire sampling volume. This may best be achieved through the use of composite samples from several containers.

The number of samples required to detect a specific difference, at a given level of probability, may be calculated:

$$\text{Number of samples} = t^2 s^2 / d^2$$

where:  $t$  = distribution of variables at given level of probability

$s^2$  = estimated variance

$d$  = size of difference to be detected

However, this requires some knowledge of inherent variation which is often unavailable. Therefore, the number of samples used for analysis is usually determined by factors such as time, economics, and convenience.

The preparation of soil samples generally includes grinding to break up aggregates. Since bark medium is much coarser than soil, grinding would interfere with many analytical objectives. However, some measures must be taken to minimize the variability in the texture of bark samples. Results from the determination of an adjusted particle size



distribution for bark samples are presented in Table X.

The coarsest particles (NBS sieve no. 4) of the unamended samples represented the smallest percentage, by weight, of the total separates. However, this fraction also had the highest standard error. The heterogeneity created by the inclusion of these coarse particles in bark samples interferes with many basic analytical objectives. Therefore, it is apparent that some adjustments are necessary.

Jackson (108) has suggested that the largest particles in a soil sample should constitute 0.001 or less of the minimum sample volume. However, the particle size range for bark medium is much wider than for soils. Therefore, Jackson's criterion is not totally suitable. While it is essential to maintain a representative distribution of particles in bark samples, heterogeneity must be reduced.

The particle size distribution of the amended samples in which the coarse particles (NBS sieve no. 4) were removed demonstrates its effect on increased homogeneity. This may be attributed to a more uniform distribution of particles, and reduced variability within each fraction.

Without question the exclusion of the coarse particles from bark samples will influence the results of any analytical determination. However, these particles contribute primarily to the bulk matrix of the media and their reactivity is limited. Therefore, the increased accuracy with which analyses may be conducted justifies such adjustments in the particle size distribution of the bark samples.

Table X. Determination of an adjusted particle size distribution for samples of southern pine bark.<sup>1</sup>

	Particle distribution (%/wt)			
	NBS sieve no.			Receiver pan
	4	10	20	
	Openings (mm)			
	2.00	0.84	0.60	< 0.42
Unamended samples	16.11	31.00	25.06	27.75
Std. error	0.286	0.06	0.068	0.1
Amended samples	--	45.46	31.39	23.14
Std. error	--	0.054	0.05	0.078
Percentage adjustments		+14.46	+6.26	-4.61

<sup>1</sup>Means of 25 samples.

The percentage adjustments best indicate the relative changes in each of the fractions. The increased percentage of particles retained on the no. 2 and 3 mesh sieves may be attributed to the redistribution of total separates. Because these particles are less variable in size, a more uniform texture results as indicated by a reduction in the standard error of these two fractions.

The decreased percentage of particles retained on the receiver pan is a further indication of the redistribution of separates. This limited reduction in highly reactive particles may be considered as partial compensation for the exclusion of the coarser, less reactive particles from the bark samples. However, this is not a proportionate exchange.

Since both physical and chemical analyses are conducted on a volume basis, the effect of particle size distribution on bulk density must also be considered. By removing the coarse bark particles (NBS sieve no. 4), the sample bulk density decreased from 0.274 to 0.262 g/cc. This may be attributed to the relatively high specific gravity of the coarser particles. However, the bulk density of these adjusted samples was less variable which increased sample homogeneity. These data further demonstrate the importance of sample preparation on the interpretation of analytical results.

Compaction is another factor which can affect sample bulk density. Although the amount of compaction used for bark samples should simulate actual growing conditions,

this is not always possible. Therefore, uniformity is stressed. The "tapping" method is useful; however, the procedure is largely qualitative and only through repeated use can samples be standardized.

Based on the data from this experiment, it may be concluded that the interpretation of analytical results will not be significantly affected by adjustments to the particle size distribution and bulk density of bark samples. The increased homogeneity which results from the removal of the coarser particles reduces variability both within and between samples without interfering with basic analytical objectives.

#### The Chemical Properties of Southern Pine Bark:

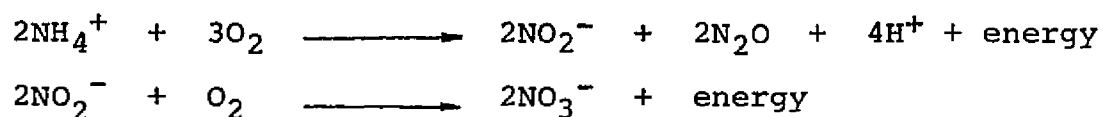
The chemical properties of soilless growing media provide the link between fertility and those physical properties previously discussed. The aspects of solution and solid phase chemistry which maintain suitable concentrations of nutrients in the growing medium are essential factors in plant growth. Therefore, it is necessary to characterize the basic chemical properties of bark medium in order to understand its effect on substrate fertility.

Ash is a primary chemical constituent of southern pine bark. The total mineral analysis from this ash may be used as a background for the interpretation of substrate fertility and as a means of assessing the potential nutrient "availability" of bark media. The results from the total mineral analysis of southern pine bark are presented in

Table XI.

Most of the N in pine bark is combined in complex protein molecules. Before this N can become "available" for plant uptake, partial decomposition must occur. During this process, the C to N bond of the basic amine groups is broken and the adsorption of  $H^+$  ions results in the formation of  $NH_4^+$ . This ammonification process is dependent on microbes and the enzymes which they produce. Since most microbes can decompose organic matter, ammonification takes place more rapidly when conditions favor microbial activity.

The decomposition of pine bark also involves a two-stage oxidation process in which  $NH_3^+$  is oxidized to  $NO_2^-$  and then to  $NO_3^-$ :



Almost any microbe can carry out ammonification, but only a few specific ones are involved in the formation of  $NO_3^-$ . Nitrosomonas sp. (and a few others) oxidize  $NH_3^+$  to  $NO_2^-$ , and Nitrobacter sp. oxidize  $NO_2^-$  to  $NO_3^-$ . Significant amounts of  $NO_2^-$  rarely occur in growing media because it is immediately oxidized to  $NO_3^-$ . However, in the absence of these specific microorganisms, a toxic accumulation of  $NO_2^-$  could result.

Although microbial activity determines whether  $NH_4^+$  and  $NO_3^-$  will be mineralized or immobilized, the principal factor involved here is the C:N ratio. Generally, a C:N of 32:1 is considered the break-even point for the

Table XI. Total mineral analysis of southern pine bark.<sup>1</sup>

N (%)	P (%)	K (%)	Element		Fe (ppm)	Zn (ppm)	Mn (ppm)
			Ca (%)	Mg (%)			
0.436 <sup>1</sup>	0.059	0.367	0.442	0.053	721	124	66
0.009 <sup>2</sup>	0.001	0.002	0.002	0.001	3.802	2.444	1.631
0.045 <sup>3</sup>	0.005	0.010	0.010	0.005	19.01	12.22	8.155

<sup>1</sup>Means of 25 samples.

<sup>2</sup>Standard error.

<sup>3</sup>Standard deviation.

decomposition or organic matter. Therefore, due to the relatively wide C:N of pine bark, most of the  $\text{NH}_4^+$  and  $\text{NO}_3^-$  formed during decomposition is immobilized by microorganisms. Other factors which affect the mineralization and immobilization of N include water, oxygen, pH, and temperature.

The determination of total N in pine bark agreed well with previously reported values. However, since  $\text{NO}_3^-$  and fixed  $\text{NH}_4^+$  are not estimated by the Kjeldahl method, it is difficult to interpret the potential "availability" of N.

Most containerized floricultural crops require relatively high rates of N fertility. Therefore, supplemental applications of N will be required. Although several types of N fertilizers may be used for supplemental application, three principal sources are used for most floricultural crops.

Ammonium nitrate ( $\text{NH}_4\text{NO}_3$ ) contains approximately 33 percent N and is perhaps the most widely used of all N fertilizers. The high solubility and balance between  $\text{NH}_4^+$  and  $\text{NO}_3^-$  make  $\text{NH}_4\text{NO}_3$  an excellent N source for liquid fertilization.

Potassium nitrate ( $\text{KNO}_3$ ) is another important N source because it provides both K and  $\text{NO}_3^-$ . The analysis of this material is approximately 13-0-45. Potassium nitrate finds its greatest use on crops which respond to a delayed application of both K and N. The solubility of this material also lends itself well to liquid fertilization.

Calcium nitrate  $\text{Ca}(\text{NO}_3)_2$  supplies approximately 15.5 percent N in the  $\text{NO}_3^-$  form. Calcium nitrate is an important

N source for floricultural crops because it provides Ca which is often required late in the growing season. As another soluble form of N, it too may be used in liquid fertilization.

The total P content of pine bark may be classed, generally, as organic or inorganic, depending on the type of compound in which it occurs. Inorganic P occurs in combinations with Fe, Al, Ca, F and other elements. Organic P occurs in phospholipids, nucleic acids and other organic molecules.

Most of the organic P is tightly held by at least one covalent bond. These are primarily C-O-P bonds which are broken as the organic molecule decomposes. When this occurs, P is mineralized and made "available" for plant uptake. Generally, the stability of these P bonds is greater under acid conditions. Therefore, the increased mineralization of organic P is associated with rises in pH.

The mineralization of organic P is also related to concentrations of C. Generally, a C:P ratio of 200:1 or less will result in the mineralization of P. However, a C:P ratio of 300:1 or greater will result in immobilization. This is largely attributable to the effects of microbial activity on the decomposition of organic molecules containing P.

Plants absorb P, primarily, as the orthophosphate ions  $\text{H}_2\text{PO}_4^-$  and  $\text{HPO}_4^{--}$ . The concentration of these various phosphate ions in solution is largely pH dependent. The  $\text{H}_2\text{PO}_4^-$  ion is favored under acidic conditions whereas the



$\text{HPO}_4^{--}$  ion is favored above pH 7.0. This may be attributed to the formation of the insoluble phosphates of Fe and Al under acidic conditions, and the formation of the insoluble phosphates of Ca and Mg at pH values above 7.0. Generally maximum P "availability" occurs within the pH range of 5.5 to 7.0.

The determination of total P in pine bark also agreed well with previously reported values. However, it was apparent that supplemental P will be necessary for optimum plant growth. Phosphorus may be supplied to plants in various forms. However, solubility is of primary importance. Ordinary superphosphate contains approximately 20 percent  $\text{P}_2\text{O}_5$  (7 percent P) and is a widely used source of supplemental P. In addition, ordinary superphosphate contains about 8 percent sulfur as  $\text{CaSO}_4$ . Ordinary superphosphate is approximately 90 percent "available" to plants; however, it is relatively slow releasing. This material is an excellent P source and is frequently added (pre-plant) to growing media.

Phosphoric acid ( $\text{H}_3\text{PO}_4$ ) contains approximately 55 percent  $\text{P}_2\text{O}_5$  (24 percent P) and is frequently used in the preparation of liquid fertilizer solutions. Although the relatively high analysis of this material is attractive,  $\text{H}_3\text{PO}_4$  may result in the precipitation of secondary P and Ca compounds.

Potassium is absorbed by plants in larger amounts than any other mineral element, except N. However, a specific role for K in plants is, as yet, unknown. The highest

concentrations of K are found in the meristematic regions of the plant. Therefore, the K level of pine bark may be expected to be relatively high in association with cambial activity.

The organic and inorganic salts of K are released when they change from a nonexchangeable to an exchangeable form. This involves the decomposition of bark particles in which K, fixed in organic molecules, is brought into solution. Most of these K compounds are readily soluble, and highly ionized in solution.

Plants absorb potassium in the form of  $K^+$  ions. The positive charges of these cations help to maintain electrical neutrality in both the growing medium and the plant by balancing the negative charges of various anions.

Exchangeable K occurs as hydrated K ions attracted to the negatively charged exchange sites of bark particles. These hydrated K ions are held less tightly than other macro-nutrient cations. Therefore, they are readily exchangeable because of the relatively low energy of attraction.

Generally, the amount of "available" K is less under acid conditions than alkaline conditions. This may be attributed to the increased concentration of  $H^+$  ions and reduced concentration of  $K^+$  ions on the exchange sites, and in solution, at lower pH values. Although the pH of a growing medium may be neutralized by liming, this adds  $Ca^{++}$  and often  $Mg^{++}$  ions to the medium, but not  $K^+$ . Under some circumstances, the "available" K supply may be increased for a time by liming; however, the reverse will

generally occur.

The determination of total K in pine bark would indicate that supplemental K applications will be necessary for optimum plant growth. Although several K sources are available (i.e.,  $K_2SO_4$ , KCl and potassium magnesium sulfate), potassium nitrate ( $KNO_3$ ) is the most commonly used material for floricultural crops.

The importance of  $KNO_3$  from the standpoint of N has already been discussed. However, its 44 percent  $K_2O$  content makes it an equally important source of K. Although the production cost of this material is relatively high, its solubility and subsequent "availability" is excellent for use in liquid fertilizer solutions.

Calcium occurs in pine bark primarily as a constituent of cell walls in the form of calcium pectate. Upon its release, as the result of decomposition of these organic molecules, Ca becomes "available" for plant uptake.

Calcium is absorbed by plants as the  $Ca^{++}$  ion. When these  $Ca^{++}$  ions are released into bulk solution, they may be lost to leaching, absorbed by living organisms, adsorbed onto exchange sites, or reprecipitated as a secondary Ca compound. Like other cations, the exchangeable and solution forms of Ca are ever changing. If the activity of Ca in the solution phase is decreased, replacement will occur from the adsorbed phase. Conversely, if the activity of Ca in the solution phase is increased, there will be a shift in equilibrium in the opposite direction, with subsequent adsorption of some of the Ca by the exchange complex.

The heavy irrigation regimes used in association with soilless growing media frequently result in reduced pH values. This may be attributed to the removal of Ca due to excessive leaching. As water containing dissolved  $\text{CO}_2$  percolates through the growing medium, the carbonic acid formed displaces Ca on the exchange complex. Considerable percolation of such water results in a gradual reduction in pH.

Although the total concentration of Ca in the pine bark was relatively high, it is apparent that supplemental applications will be required for use by the plant and for pH adjustments. Supplemental applications of Ca are usually derived from three basic sources: calcium nitrate; dolomitic lime; and gypsum. The advantages of  $\text{Ca}(\text{NO}_3)_2$  have been previously discussed.

Dolomitic limestone ( $\text{CaCO}_3 \cdot \text{MgCO}_3$ ) is used to supply Ca and Mg as well as to adjust pH. This material is frequently used in the preparation of soilless growing media.

Gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) is an excellent Ca source when adjustments to pH are not required. This material may also be used to increase the stability of a growing medium and to replace Na on the exchange complex.

In plants, Mg is primarily a constituent of the chlorophyll molecule. Therefore, the Mg content of pine bark would not be expected to be significant. However, the decomposition of bark particles does result in a limited release of Mg into the bulk solution. Similar to Ca, this Mg is subject to several factors. Upon release

it may be: lost due to leaching; absorbed by living organisms, adsorbed onto exchange sites; or reprecipitated as a secondary Mg compound.

Magnesium is absorbed by plants as the  $Mg^{++}$  ion. Like  $Ca^{++}$  and  $K^+$  the adsorption of  $Mg^{++}$  takes place largely from the soil solution and by contact exchange. "Available" Mg occurs in both the exchangeable and water soluble forms. However, the absorption of Mg by the plant is dependent on several factors. These include: the amount of Mg present, the degree of Mg saturation, the presence of other exchangeable ions, and the nature of the exchange sites.

The determination of total Mg in pine bark indicates relatively low concentrations. Therefore, it may be concluded that Mg presents more of a potential for deficiency than the other macronutrients examined in this study.

Perhaps the most important supplemental source of Mg is dolomitic lime ( $CaCO_3 \cdot MgCO_3$ ). This material is used to supply Ca and Mg while the carbonate portion neutralized pH. For a more rapid response, magnesium sulphate ( $MgSO_4 \cdot 7H_2O$ ) may also be used. With a Mg content of approximately 9.6 percent, this material is an excellent supplemental form. Other sources of Mg include potassium-magnesium sulphate, magnesia, and manures.

Micronutrients occur in pine bark as the constituents of various organic compounds. However, their concentrations are relatively low. As bark particles decompose, they release these micronutrients into solution. Unlike the situation with macronutrients discussed, this release represents

the primary micronutrient source in pine bark growing medium.

Iron is absorbed by plants primarily as the ferrous ion ( $\text{Fe}^{++}$ ). Many of the compounds of ferrous iron have low solubilities, but ferric iron ( $\text{Fe}^{+++}$ ) compounds are even less soluble. The oxidation and reduction of the various forms of Fe is dependent on several factors.

When oxygen is excluded from the growing medium, such as in frequent irrigation, ferric iron is reduced to the ferrous form. However, under well drained conditions, these ferrous compounds are rapidly oxidized to the ferric state. Therefore, it may be concluded that the status of "available" Fe is very dynamic in bark media. Furthermore, reduction of Fe is also hastened by the presence of organic matter. This may be partially attributed to the release of  $\text{H}^+$  ions during decomposition, and to an increased water-holding capacity.

The solubilities of ferric and ferrous iron are much lower at high pH than at low pH. Both  $\text{Fe}(\text{OH})_3$  and  $\text{Fe}(\text{OH})_2$  have low solubilities and can be precipitated at high pH because  $\text{OH}^-$  ions become more abundant when pH rises. Other Fe compounds also become less soluble at higher pH values. The precipitation of previously "available" Fe is one of the greatest problems in liming a growing medium. Therefore, supplemental forms are often required.

The determination of total Fe in pine bark indicates the potential for sufficient release for plant growth. However, due to the oxidation and reduction of this element and the relatively high pH of a growing medium, supplemental

application is usually necessary.

Iron may be supplied to floricultural crops by applying Fe compounds to the growing medium or directly to the foliage in an aqueous spray. Fritted iron and iron chelates are perhaps the most commonly used materials applied to the growing medium, while iron sulfate ( $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ) is the inorganic salt most commonly used in foliar sprays.

Manganese occurs in a growing medium in three valence states:  $\text{Mn}^{++}$  which is present as an adsorbed cation or in the bulk solution;  $\text{Mn}^{+++}$  which exists as a highly reactive oxide ( $\text{Mn}_2\text{O}_3$ ); and  $\text{Mn}^{++++}$  which exists as a very inert oxide ( $\text{MnO}_2$ ). These three forms of Mn exist in equilibrium with one another in the growing medium.

The highly stable  $\text{MnO}_2$  is most likely to occur at pH values above 8.0. The trivalent form of Mn is favored by pH values near neutrality, whereas the divalent form is found under acid conditions. The oxidation potential for the conversion of  $\text{Mn}^{++}$  to  $\text{MnO}_2$  is a linear function of pH between the values of 3.2 to 8.0. Whether the pH or the oxidation status of the growing medium is more closely related to Mn "availability" has not been established.

Organic matter and moisture also influence the "availability" of Mn. Generally large amounts of organic matter at pH values near 7.0 result in Mn deficiencies. This is attributed to the formation of insoluble complexes with  $\text{Mn}^{++}$  by certain types of organic matter. Furthermore, the increased water-holding capacity resulting from high concentrations of organic matter also reduces the "availability"

of Mn. Because most containerized floricultural crops are grown at relatively high pH and moisture conditions, the determination of total Mn in pine bark is not a good indication of its potential "availability." Therefore, it may be concluded that supplemental applications of Mn will be necessary.

Similar to Fe, Mn may be applied to the growing medium or directly to the foliage of actively growing plants. Manganese sulfate ( $\text{MnSO}_4 \cdot 3\text{H}_2\text{O}$ ) contains approximately 25 percent Mn and is perhaps the most commonly used source for foliar applications. Manganese frits and chelates are primarily used for applications to the growing medium and their Mn content varies widely.

The  $\text{Zn}^{++}$  ion is strongly adsorbed on the exchange complex of bark particles. This strong attraction of positively charged  $\text{Zn}^{++}$  ions to negatively charged exchange sites is one of the primary factors which limits Zn "availability."

The level of P in a growing medium also influences Zn "availability." High phosphate levels have been shown to reduce the solubility of Zn. This is generally attributed to the precipitation of zinc phosphate. However, other factors are involved. These include high concentrations of organic matter and the effect of microbial activity on Zn immobilization.

Zinc is generally more "available" to plants in an acid medium than an alkaline medium. As a rule, most Zn deficiencies occur within a pH range of 6.0 to 8.0. This may be attributed to the adsorption of Zn by the carbonates



of Ca and Mg. The resulting precipitation of carbonates and hydroxides can reduce "available" Zn to deficient levels.

The determination of the total concentration of Zn in pine bark indicates the need for supplemental application. Similar to Fe and Mn, the supplemental application of Zn may be made to the growing medium or to the foliage of actively growing plants. Zinc sulfate ( $\text{ZnSO}_4 \cdot \text{H}_2\text{O}$ ) and Zn frits and chelates represent the principal sources of these Zn fertilizers.

The interpretation of analytical values regarding the "availability" of nutrient elements is dependent on several factors. Perhaps the most important of these are: substrate:extractant ratio; type of extractant used; and the length of extraction time.

Recently, the Floriculture Working Group of the American Society of Horticultural Science proposed the saturated media extract method for the analysis of soilless growing media. However, this method has not proven satisfactory for bark media. Because each sample is prepared from a different quantity of extractant (depending on its absorption characteristics) analytical values must be reported with an accompanying value for the saturation percentage. Therefore, the interpretation of these values must be based on the interaction between the concentration of extracted nutrients and the amount of extractant used.

Although fixed substrate:extractant ratios do not account for textural differences in growing media, they have been found to be a more reliable means of estimating

the "availability" of nutrients in bark media. Due to the hydrophobic nature of dried bark, a relatively large quantity of extractant is required for "wetting." Once this has occurred, bark readily absorbs the extractant. Because this initial "wetting" process requires a relatively long period of time, the development of a saturated paste is not feasible. Therefore, wider substrate:extractant ratios are more suitable. Furthermore, by standardizing the quantity of extractant used, analytical values reflect the concentrations of extracted nutrients without variation in the saturation percentage.

Several substrate:extractant ratios were investigated previous to this experiment (data not included). Generally as these ratios became more dilute, handling was easier and analytical values became more stable. Based on these observations, a 1:4 v/v substrate:extractant ratio was used for the extraction of nutrients from the bark medium.

The selection of an analytical system for substrate analysis should be based on its ability to extract nutrients related to plant response. However, since no one system is suitable for all types of growing media or all elements, various types have been developed. Both solvent and exchange extractants have been proposed for the analysis of bark media. These differ, primarily, in their method of extraction.

The Spurway (214) test has long been considered the standard soil test for most horticultural crops. This method uses dilute acetic acid (0.025 N HOAc) to remove

water soluble nutrients from the bulk solution. However, the reserve or exchangeable nutrients are not measured by this method.

The L.S.U. analytical system (39) utilizes neutral normal ammonium acetate ( $1.0 \text{ N } \text{NH}_4\text{OAc}$  pH 7.0) for the extraction of cations. This buffered salt solution removes exchangeable as well as water soluble nutrients from the growing medium. However, the effectiveness of this solution may be limited under extremely acid or alkaline conditions, and in the presence of large quantities of organic matter.

The L.S.U. system also utilizes a modified Bray solution ( $0.1 \text{ N } \text{HCl} + 0.03 \text{ N } \text{NH}_4\text{F}$ ) for the extraction of P from the growing medium. However, this method may over-estimate the quantity of "available" P from various organic sources.

Melich's (151) double acid solution (equal parts of  $0.025 \text{ N } \text{HCl}$  and  $0.05 \text{ N } \text{H}_2\text{SO}_4$ ) is the predominant soil extractant used in analytical systems throughout the southeastern U. S. The combination of strong oxidizing and reducing acids in the double acid solution also removes exchangeable as well as water soluble nutrients from the medium. However, the double acid solution is not suitable for all media types.

Based on the composition and action of these extractants, it is apparent that each will vary in the type and quantity of nutrients it removes from the growing medium, and the interpretation of nutrient "availability" must

take this into consideration.

The extraction of nutrients from a growing medium is also dependent on the length of time that the medium is in contact with the extractant. As previously discussed, dried bark requires a relatively long period of time for initial saturation. However, once this has occurred, the quantity and nature of nutrients extracted becomes a function of the extraction time. The interpretation of nutrient "availability" must be based on the interrelationship between the extractant and the length of the extraction time.

As the extraction time was lengthened, the quantity of P, K, Ca and Mg removed by each of the three extractants increased until equilibrium was reached. At that point, the quantity of nutrients removed leveled off even though the length of the extraction time continued to increase. Under these conditions, a second degree regression equation describes the relationship between extraction times and the quantity of P, K, Ca and Mg removed by each of the three extractants (Tables XII, XIII, XIV).

Phosphorus, potassium, calcium and magnesium occur in the bulk solution of bark media, primarily, as the result of the decomposition of organic molecules and the addition of fertilizers. If the activity of these nutrients in the solution phase is decreased, replacement will generally occur from the adsorbed phase. However, if activity in the solution phase is increased, a shift in equilibrium in the opposite direction usually occurs. Since plants

Table XII. Second degree equations describing the effect of extraction times<sup>1</sup> on the quantity of "available" P, K, Ca and Mg in pine bark growing medium, as determined by the modified Spurway method.

Element	Equation <sup>2</sup>	R <sup>2</sup>
P (ppm)	$6.73 + 1.48X - 0.045X^2$	0.670
K (ppm)	$10.89 + 1.32X - 0.040X^2$	0.665
Ca (ppm)	$72.11 + 8.56X - 0.255X^2$	0.885
Mg (ppm)	$5.62 + 2.30X - 0.067X^2$	0.925

<sup>1</sup>Range of extraction times 1-24 hrs.

<sup>2</sup>All coefficients significant at the .05 level of probability.

Table XIII. Second degree equations describing the effect of extraction times<sup>1</sup> on the quantity of "available" P, K, Ca and Mg in pine bark growing medium as determined by the modified LSU method.

Element	Equation <sup>2</sup>	R <sup>2</sup>
P (ppm)	$11.19 + 5.450X - 0.15X^2$	0.975
K (meq/100 cc)	$0.12 + 0.043X - 0.01X^2$	0.891
Ca (meq/100 cc)	$3.99 + 0.831X - 0.024X^2$	0.930
Mg (meq/100 cc)	$1.10 + 0.132X - 0.0039X^2$	0.945

<sup>1</sup>Range of extraction times 1-24 hrs.

<sup>2</sup>All coefficients significant at the .05 level of probability.

Table XIV. Second degree equations describing the effect of extraction times<sup>1</sup> on the quantity of "available" P, K, Ca and Mg in pine bark growing medium as determined by the modified double acid method.

Element	Equation <sup>2</sup>	R <sup>2</sup>
P (ppm)	$27.16 + 4.350X - 0.12X^2$	0.922
K (meq/100 cc)	$0.20 + 0.041X - 0.0012X^2$	0.919
Ca (meq/100 cc)	$9.44 + 0.212X - 0.006X^2$	0.937
Mg (meq/100 cc)	$1.06 + 0.126X - 0.003X^2$	0.969

<sup>1</sup>Range of extraction times 1-24 hrs.

<sup>2</sup>All coefficients significant at the .05 level of probability.

absorb P, K, Ca and Mg largely from the bulk solution of a growing medium, a measure of these nutrients in solution phase provides a good indication of their "availability."

The solvent action of the HOAc used in the Spurway test is designed to simulate the soil solution process. This involves the extraction of water soluble (and some weakly adsorbed) nutrients from the bulk solution of the growing medium. Therefore, the quantity of P, K, Ca and Mg removed from the bark medium by HOAc extraction would be expected to be less than the two exchange extractants.

Neutral normal ammonium acetate and the double acid solution remove exchangeable as well as bulk solution ions from the growing medium. However, the quantity of exchangeable nutrients removed is based on their composition and action.

Neutral normal ammonium acetate is not generally considered a standard extractant for P. Therefore, a separate P extraction is usually conducted in the L.S.U. analytical system where  $\text{NH}_4\text{OAc}$  is used for cation extraction, although several such P extractants have been proposed for soilless growing media. However, the modified Bray solution ( $0.1 \text{ N HCl} + 0.03 \text{ NH}_4\text{F}$ ) is commonly used in the L.S.U. system. The HCl component of this solution is designed to extract acid soluble Ca, Fe and Al phosphates, whereas the  $\text{NH}_4\text{F}$  component complexes  $\text{Al}^{+++}$  and  $\text{Fe}^{+++}$  in acid solution. Since neither Fe or Al are particularly abundant in bark media, it is likely that calcium phosphate is the predominant P form extracted.

The net negative charge on bark particles is due primarily to the presence of carboxylic and phenolic groups. As these organic compounds ionize they produce a negative charge to which cations become covalently bound. The extent of this ionization, however, is largely pH dependent. Generally, the pH range between 7.0-9.0 results in the greatest ionization of carboxylic and phenolic groups. The determination of "available" K, Ca and Mg is thus contingent on the pH of the growing medium and the pH of the extractant used.

If a growing medium is extracted with a neutral, unbuffered salt solution, only the cations held at active exchange sites will be removed. However, if a buffered solution is used (i.e., 1.0  $\underline{N}$   $\text{NH}_4\text{OAc}$  pH 7.0), the quantity of nutrients removed will be influenced by the pH of the extractant. The amount of K, Ca and Mg extracted from the bark medium by 1.0  $\underline{N}$   $\text{NH}_4\text{OAc}$  pH 7.0 represents ions from the bulk solution and those adsorbed to organic radicals. However, the quantity of exchangeable cations is modified by the effect of pH on the degree of ionization of the carboxylic and phenolic groups.

Phosphorus extraction with the double acid solution primarily removes acid soluble forms of phosphate. However, as the  $\text{HCl}$  and  $\text{H}_2\text{SO}_4$  components dissociate,  $\text{Cl}^-$  and  $\text{SO}_4^-$  anions are released. This results in the replacement of  $\text{H}_2\text{PO}_4^-$  and  $\text{H}_2\text{PO}_4^{--}$  on the exchange complex. Therefore, the quantity of P removed from the bark medium by double acid extraction would be expected to be greater than that

extracted by the modified Bray solution.

This dissociation of HCl and H<sub>2</sub>SO<sub>4</sub> also results in the release of H<sup>+</sup> ions. Therefore, when a growing medium is extracted with the double acid solution, cations are displaced from organic radicals by the preferential adsorption of H<sup>+</sup>. Furthermore, the strong oxidizing and reducing potentials of these two acids increases the quantity of cations released. Since H<sup>+</sup> ions are more readily adsorbed than NH<sub>4</sub><sup>+</sup> ions, the quantity of K, Ca and Mg extracted from the bark medium would be expected to be greater for the double acid solution than neutral normal ammonium acetate.

Based on the data from this experiment, it may be concluded that as extraction times were lengthened, the quantity of P, K, Ca and Mg extracted from the bark medium increased for each of the three extractants. This may be attributed to the increased absorption and adsorption of the extracting solutions. The quantity and type of nutrients removed from the bark medium also varied according to the composition and action of each of the three extractants. Therefore, it is essential to consider these factors in the interpretation of analytical results.

Durability is a term often used to describe the effects of factors which influence the decomposition of organic matter. These include: the percentage of lignin, C, N, and the C:N ratio. Results from the determination of these properties in southern pine bark are presented in Table XV.

Lignin is an important constituent of pine bark



Table XV. Durability properties of southern pine bark.<sup>1</sup>

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Lignin (%/wt)	53.33
Std. error	0.06
Carbon (%/wt)	49.07
Std. error	0.31
Nitrogen (%/wt)	0.40
Std. Error	0.01
C:N	122.982
Std. Error	7.87

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<sup>1</sup>Means of 25 samples.

because it provides some resistance to decomposition. This may be attributed to the inability of most microorganisms to use lignin as an energy source. Because lignification occurs most extensively in secondary tissue, the lignin content of bark is relatively high. Therefore, it may be concluded that the durability of bark medium is increased due to its high lignin content.

The percentages of C and N largely determine the amount of microbial activity involved in decomposition. Microorganisms use C as an energy source, and, thus, a relatively high C content stimulates an increase in the number of heterotrophic organisms. These microorganisms also depend upon the N present to synthesize proteins and other cellular constituents. Although these two factors are important from the standpoint of decomposition, it is the C:N ratio which most affects durability. If the C:N ratio is wide, microorganism activity is reduced and durability is increased. However, under these conditions, microorganisms utilize other potential N sources. Because they can compete for N more effectively than plants, N depletion may occur. As the C energy supply is diminished through decomposition, the C:N ratio will narrow and ultimately a new equilibrium will be reached.

Although the C:N ratio is an important characteristic which influences N availability and the durability of growing media, it is not a primary factor in the production of containerized floricultural crops. The use of relatively high levels of N fertility and the short growing period

of most crops largely offsets the problem of N depletion. Therefore, it may be concluded that the high lignin content and wide C:N ratio of pine bark growing media provide excellent properties of durability and do not interfere with plant nutrition.

Several factors must be considered in the interpretation of CEC values for soilless growing media. Perhaps the most fundamental of these is the nature and origin of the exchange complex. The net charge on bark particles is due primarily to carboxylic and phenolic groups which result in the covalent bond of  $H^+$  and other ions. Also, ultrastructure studies have shown that bark particles have a relatively large surface area. This combination of characteristics gives rise to an exchange capacity more than twice that of soils on a weight basis.

The determination of CEC is largely contingent on the pH of the growing medium and the pH of the index extractant used. If a growing medium is extracted with a neutral, unbuffered salt, only the cations held at active exchange sites will be replaced. However, if a buffered solution is used, the quantity of cations replaced will be indicative of the pH of the index extractant. Therefore, the CEC of a growing medium is a continuous function of pH, with CEC values increasing as pH increases.

Because most soilless growing media are limed to a relatively neutral pH, the use of a buffered index solution would not be expected to yield CEC values which significantly differ from an unbuffered solution. That is to say

that the quantity of replaced cations would not significantly differ between the existing pH of the growing medium and the adjusted pH of the buffered index extractant.

The significant difference which occurred between the two buffered index extractants used in this experiment (Table XVI) may be attributed to several factors. Highly buffered, normal salt solutions of metallic cations, which form strong bases, tend to measure greater amounts of exchange capacity than those which form weak bases. This may be attributed to the relatively large quantity of metallic cations, compared to  $H^+$ , in the salt solution. Further, normal acetate salts of monovalent ions usually dissolve only small amounts of organic matter in solutions with a moderately high pH. Therefore, the 1.0 N  $BaCl_2$  pH 8.0 measured a significantly higher CEC than 1.0 N  $NH_4OAc$  pH 7.0.

The length of the extraction time is another important factor which must be considered in the interpretation of CEC values. The determination of CEC is dependent on the saturation of the growing medium. Therefore, the length of time required for extraction with the index solution is contingent upon the physical and chemical nature of the media and its exchange sites. Most soils are extracted for 5-10 minutes; however, due to the hydrophobic characteristic of dried bark samples, this length of time is not suitable.

The CEC values obtained in this experiment significantly increased as the extraction time was lengthened from

Table XVI. Determination of the CEC of southern pine bark.<sup>1</sup>

Index extractant	Extraction time			
	2 hr.		24 hr.	
	Media:extractant ratio (v/v)			
	1:2	1:4	1:2	1:4
1.0 N BaCl <sub>2</sub> pH 8.0	12.49 <sup>2</sup>	12.98	13.56	14.02
Std. error <sup>3</sup>	0.33	0.24	0.16	0.15
1.0 N NH <sub>4</sub> OAc pH 7.0	10.41	11.44	11.34	12.11
Std. error <sup>3</sup>	0.18	0.34	0.25	0.33

<sup>1</sup>Means of 6 samples.<sup>2</sup>Milliequivalents/100 cc.<sup>3</sup>Based on between sample variation.

Table XVII. Analysis of variance for the determination of the CEC of southern pine bark.

Source	Degrees of freedom	Sum of squares	F
Extractant	1	45.105	107.78*
Time	1	10.314	24.65*
Ratio	1	5.692	13.60*
Extractant X Time	1	0.189	0.45
Extractant X Ratio	1	0.561	1.34
Time X Ratio	1	0.058	0.14
Extractant X Time X Ratio	1	0.041	0.10
Error	40	16.740	-
Total	47	78.700	-

\*Significant at the 0.05 level of probability.

2 to 24 hours. This increase may be attributed to the "wetting" of the bark particles which resulted in the increased adsorption of the index extractant. Furthermore, variability in CEC values was reduced at the longer extraction time.

Similar to the determination of the "availability" of nutrient elements in soilless growing media, the saturated media extract method has been proposed for the measurement of exchange capacity. As previously stated, this procedure has not proven satisfactory for use with pine bark. Therefore, fixed substrate:extractant ratios are recommended. Generally, as substrate suspensions became more dilute, CEC values increased, as indicated by the significant increase in CEC values from the 1:2 to the 1:4 (v/v) substrate:extractant ratio. It may further be noted that the more dilute suspensions required a longer period of time to equilibrate, as indicated by the reduced variability at the longer extraction time.

Based on these data, some generalizations may be made regarding the determination of the CEC of pine bark growing media:

1. Highly buffered, normal salt solutions of metallic cations, which form strong bases, will tend to measure greater amounts of exchange capacity than those that form weak bases.
2. As the extraction time lengthens, CEC values will tend to increase and become less variable.
3. As substrate:extractant ratios become more

dilute, CEC values will tend to increase and become less variable. However, a longer equilibration time will be required.

Acidity in southern pine bark results primarily from the covalent bonding and subsequent dissociation of  $H^+$  ions by carboxylic and phenolic groups. The amount of active  $H^+$  ion activity is generally stated in terms of pH. Because the pH measurement varies widely with the method of determination used, several factors must be considered in the interpretation of analytical results. These variables include: the presence of soluble salts; the substrate:solution ratio; and the equilibration time. The results from the effect of these factors on the determination of the pH of southern pine bark are presented in Table XVIII.

The presence of soluble salts in pine bark growing medium may be accounted for by the decomposition of bark particles and the addition of fertilizers. The cations of these salts displace adsorbed H and Al resulting in increased acidity. This condition is further intensified when acid-forming fertilizers (i.e.,  $NH_4NO_3$ ) are used.

To correct for the acidifying effects of soluble salts, pH may be determined in a salt solution (i.e.,  $CaCl_2$ ) as opposed to water. Although such pH values are lower than those measured with water, the salt content of the bulk solution is negligible when contrasted with that of the added salt solution. This means that the salt concentration of the bulk solution will have little effect on the pH measured in the added salt solution

suspension. Therefore, this is a more precise estimate of the acidity of a growing medium than that measured in a water suspension.

Based on the data from Table XVIII, it may be concluded that the significant differences which occurred between pH determinations in 0.01 N  $\text{CaCl}_2$  and water may be attributed to the presence of excessive soluble salts. Furthermore, the reduced variability associated with 0.01 N  $\text{CaCl}_2$  indicates that the soluble salts present in pine bark significantly affect the determination of pH.

The substrate:solution ratio can also influence the measurement of pH. Generally, pH determinations on samples in the "field moist" condition may be considered the most valid in terms of the existing environment. However, the variability with which these measurements are made necessitates standardization.

Because of the difficulty in reproducing concentrated suspensions (i.e., saturated extracts), more dilute suspensions are commonly used for pH determinations. Furthermore, a greater junction potential exists in concentrated suspensions. Therefore, wider substrate:solution ratios are necessary for the precise determination of pH.

In general, the more concentrated the suspension, the lower the pH value. This may be attributed to the unequal diffusion of  $\text{K}^+$  ions (in the electrode) attracted to the negatively charged sites on the exchange complex of the bark. Therefore, the significant difference which occurred between the pH values measured in the 1:2 (v/v) and 1:4



Table XVIII. Determination of the pH of southern pine bark.<sup>1</sup>

Solution	Equilibration time			
	30 min.		24 hr.	
	Media:solution ratio (v/v)			
	1:2	1:4	1:2	1:4
Water	5.17	5.43	4.76	5.35
Std. error <sup>2</sup>	0.03	0.02	0.02	0.02
0.01 N CaCl <sub>2</sub>	4.78	5.03	4.22	4.47
Std. error <sup>2</sup>	0.02	0.01	0.01	0.00

<sup>1</sup>Means of 6 samples.<sup>2</sup>Based on between sample variation.

Table XIX. Analysis of variance for the determination of pH of southern pine bark.

Source	Degrees of freedom	Sum of squares	F
Solution	1	3.387	501.64*
Time	1	2.005	296.97*
Ratio	1	2.038	301.83*
Solution X Time	1	0.380	56.26*
Solution X Ratio	1	0.117	17.53*
Time X Ratio	1	0.000	0.00
Solution X Time X Ratio	1	0.164	24.37*
Error	40	0.270	-
Total	47	8.360	-

\*Significant at the 0.05 level of probability.

(v/v) substrate:solution ratios resulted from a significant junction potential in the concentrated suspension, and a dilution of  $H^+$  ions in solution in the dilute suspension.

The equilibration time used for the determination of pH also influences analytical values. Due to the hydrophobic nature of dried bark, an initial "wetting" period is required. This allows for the displacement of  $H^+$  from the exchange complex of the bark particles. If the equilibration time is increased, further displacement will occur resulting in decreased pH values. Therefore, it may be concluded that the significant differences which occurred between pH determinations made at 30 minutes and 24 hours may be attributed to the increased displacement of  $H^+$ .

The interaction between solutions, substrate:solution ratios and equilibration times indicates the interdependence of these factors in the determination of the pH of southern pine bark. Because most containerized floricultural crops are grown under heavy fertility regimes, the presence of soluble salts must be considered. Due to the increased junction potential in concentrated suspensions, dilute substrate:solution ratios are generally considered more suitable for pH determinations. Lastly, the hydrophobic nature of dried bark samples requires that the length of the equilibration time allow for the sufficient displacement of  $H^+$  from the exchange complex. Therefore, it may be concluded that the determination of the pH of pine bark growing media is best estimated in

0.01 N  $\text{CaCl}_2$  using a substrate:solution ratio of approximately 1:4 (v/v) and a 24-hour equilibration time.

Although pH is a good indicator of general acidity, it gives no indication of how much lime is required. Therefore, potential acidity must be considered.

The determination of exchangeable hydrogen is actually a measure of the  $\text{H}^+$  supplying power of the medium. The quantity of these protons removed in the extraction process of the analysis is reported as exchangeable hydrogen. This value may then be calculated in terms of a lime requirement.

Growing media also contain various quantities of exchangeable aluminum. When a proton acceptor (extractant) is placed in equilibrium with a system containing exchangeable aluminum, hydrolysis will occur. This results in the release of a proton from water. The net effect of this reaction is equivalent to the release of an exchangeable hydrogen ion. Therefore, the amount of exchangeable aluminum held by a growing medium must also be considered in the determination of a lime requirement.

The lime requirement of a growing medium is not only related to these factors but also to its buffer or cation exchange capacity. Those growing media with relatively high buffer capacities, if acid, generally have a high lime requirement. Therefore, adequate liming recommendations must be based on a knowledge of active and potential acidity as well as the buffer capacity of the growing medium.

The lime requirement for soils is based on the

neutralizing value of pure calcium carbonate ( $\text{CaCO}_3$ ) which has been arbitrarily established at 100%. Each meq of total exchangeable acidity to be neutralized per 100 gm of soil requires 1000 pounds of pure  $\text{CaCO}_3$  per 2,000,000 pounds of soil (or acre furrow slice 6" deep). Based on this recommendation and the data from Table XX, a lime requirement may be calculated for the pine bark growing medium:

Total exchangeable acidity = 0.231 meq/100 cc or  
0.843 meq/100 g  
(Bulk density of pine bark = 0.274 g/cc)

There are approximately 806.76 cu yds/acre furrow  
slice

Therefore;  $843 \div 806.76 = 1.04$  lbs of pure  $\text{CaCO}_3$   
required/cu yd pine bark

The CCE of dolomite = 108%

Therefore, 0.96 lb dolomite will be required/cu yd  
pine bark

Based on these calculations, it is apparent that some discrepancies exist between the lime requirement determined using total exchangeable acidity and the amount of lime actually used in the preparation of bark media. Much of this difference may be accounted for by the relatively high buffer capacity of the growing medium (approximately four times that of soil, on a weight basis). However, the principal factor involved is the method in which exchangeable aluminum and total exchangeable acidity are determined. Because the analytical method used for soils varies from that used for bark, the values are not directly comparable. Therefore, the lime requirement calculations

Table XX. Determination of exchangeable hydrogen, exchangeable aluminum and total exchangeable acidity in southern pine bark.<sup>1</sup>

	meg/100 cc	Std. error
Exchangeable H	0.152	0.003
Exchangeable Al	0.081	0.003
Total exchangeable acidity	0.231	0.004

<sup>1</sup>Means of 25 samples.

reflect these initial differences.

Based on lime requirements estimated from preliminary work with this medium, it has been established that approximately \_\_\_\_\_ lbs. of pure  $\text{CaCO}_3$  are required per cubic yard of bark. Therefore, some basic adaptations are required:

Total exchangeable acidity = 0.231 meq/100 cc or  
0.843 meq/100 g  
(Bulk density of pine bark = 0.274 g/cc)

There are approximately 764600 cc/cu yd

Assume that it requires 74.80 lbs pure  $\text{CaCO}_3$  to neutralize each meq of total exchangeable acidity/100 cc/cu yd. (Based on preliminary work)

Therefore,  $0.231 \times 74.80 = 17.28$  lbs pure  $\text{CaCO}_3$   
(= 16 lbs dolomitic lime) will be required/cu yd  
pine bark.

Based on these adaptations, it is assumed that approximately 67 lbs of  $\text{CaCO}_3$  (or 70 lbs of dolomitic lime) are required to neutralize each meq of exchangeable acidity/100 cc per cubic yard of bark media. Using these adapted figures it is possible to estimate a lime requirement which approaches that recommended for the pine bark medium. However, based on the neutralizing capabilities of  $\text{CaCO}_3$  these calculations leave considerable room for doubt as to their validity.

### Optimum Values for the Analysis of Pine Bark Growing Medium:

The relationship between nutrient levels and plant response ultimately determines the effectiveness of any analytical system. This relationship may be determined

by means of a direct correlation between foliar and substrate levels, or by estimating optimum substrate levels based on plant uptake. Each of these methods provides an excellent means of determining the reliability of analytical values.

Two principal systems have been proposed for the analysis of bark media. These differ primarily in their method of extraction. The Spurway (214) test utilizes dilute acetic acid to remove ions from the bulk solution and a relatively small quantity of ions from the exchange sites. This system simulates the natural soil solution process; however, it is not a measure of total ionic composition.

Exchange extractants, such as ammonium acetate and the double acid solution, utilize index ions to replace those on the exchange sites. Therefore, these extractants not only remove ions from the bulk solution but exchangeable ions as well.

Although these analytical methods differ in the quantity and balance of nutrients they remove from the growing medium, each has been effectively related to plant uptake (174, 238, 239). Therefore, optimum analytical values may be determined based on the relationship between nutrient uptake and supply.

Four weeks after the start of short days, the uptake of N, P, and K increased as the concentration of each of these nutrients increased in the fertilizer solutions (Table XXI). The uptake of Ca and Mg, however, decreased

Table XXI. Foliar analysis values for Euphorbia pulcherrima Willd. cv 'Annette Hegg Dark Red' four weeks after the start of short days.<sup>1</sup>

Treatment	Nutrient concentration (dry wt.)							
	N (%)	P (%)	K (%)	Ca (%)	Mg (%)	Fe (ppm)	Zn (ppm)	Mn (ppm)
1. (0.25 X)	3.89	0.306	1.49	1.52	0.429	136	75	68
2. (0.5 X)	4.21	0.441	1.80	1.43	0.428	133	79	79
3. (1.0 X)	5.06	0.521	2.77	1.33	0.396	129	80	76
4. (1.5 X)	5.95	0.673	2.91	1.31	0.364	119	80	77
CV (%)	11	7	6	7	6	12	14	15

<sup>1</sup>Means of 12 observations.

Table XXII. Foliar analysis values for Euphorbia pulcherrima Willd. cv 'Annette Hegg Dark Red' at the initiation of bract color.<sup>1</sup>

Treatment	Nutrient concentration (dry wt.)							
	N (%)	P (%)	K (%)	Ca (%)	Mg (%)	Fe (ppm)	Zn (ppm)	Mn (ppm)
1. (0.25 X)	4.21	0.398	1.99	1.37	0.391	128	71	62
2. (0.5 X)	4.71	0.451	2.33	1.30	0.372	125	71	70
3. (1.0 X)	5.20	0.529	3.22	1.23	0.331	120	73	72
4. (1.5 X)	5.96	0.678	3.37	1.18	0.321	118	76	75
CV (%)	9	11	7	9	6	13	10	12

<sup>1</sup>Means of 12 observations.



as N, P, and K increased. These data indicate the existence of a K-Ca and K-Mg antagonism, resulting in reduced Ca and Mg uptake (235).

The uptake of N, P and K continued to increase over all four fertility regimes at the initiation of bract color (Table XXII). Further reductions in the uptake of Ca and Mg may be related to increasing K levels. However, since the supply of Ca and Mg was limited, leaching was also a contributing factor. Although Ca and Mg levels were suppressed, the reported critical level for these nutrients was not reached (67). Therefore, it may be concluded that plant growth was not significantly affected.

Four weeks after the start of short days, HOAc extractable P, K and Ca increased as the result of the increasing concentrations of these nutrients in the fertilizer solutions (Table XXIII). Because this method of extraction removes primarily soluble nutrients from the growing medium, values are expressed in ppm. Since Mg was not supplied in the fertilizer solutions, but rather by initial additions to the growing medium, its concentration remained relatively constant.

Similar increases occurred over all four fertility regimes at the initiation of bract color (Table XXIV). However, reductions in Ca and Mg levels occurred as the result of leaching.

Four weeks after the start of short days, levels of P, K and Ca, as determined by the modified L.S.U. method, also increased as the result of their increasing

Table XXIII. Substrate analysis values as determined by the modified Spurway method four weeks after the start of short days.<sup>1</sup>

Treatment	pH	P (ppm)	K (ppm)	Ca (ppm)	Mg (ppm)
1. (0.25 X)	6.7	17	24	138	39
2. (0.5 X)	6.5	26	28	145	39
3. (1.0 X)	6.4	34	36	148	36
4. (1.5 X)	6.1	43	52	154	31
CV (%)	3	37	46	32	27

<sup>1</sup>Means of 12 observations.

Table XXIV. Substrate analysis values as determined by the modified Spurway method at the initiation of bract color.<sup>1</sup>

Treatment	pH	P (ppm)	K (ppm)	Ca (ppm)	Mg (ppm)
1. (0.25 X)	6.4	22	26	121	27
2. (0.5 X)	6.2	29	24	130	23
3. (1.0 X)	6.2	39	39	133	30
4. (1.5 X)	6.0	42	47	132	28
CV (%)	5	40	41	49	22

<sup>1</sup>Means of 12 observations.

concentrations in the fertilizer solutions (Table XXV). Again, Mg remained relatively constant due to its exclusion from the fertilizer solutions. Since  $\text{NH}_4\text{OAc}$  removes exchangeable as well as bulk solution ions, values for K, Ca and Mg are expressed in milliequivalents per 100 cc of growing medium.

The levels of P, K and Ca, as determined by the modified L.S.U. method, also increased over all four fertility regimes at the initiation of bract color (Table XXVI). However, Ca and Mg levels again decreased due to leaching.

Four weeks after the start of short days double acid extractable P, K and Ca also increased over the four fertility regimes while Mg levels remained relatively stable (Table XXVII). Since the double acid solution is also an exchange extractant, values for K, Ca and Mg are expressed in milliequivalents per 100 cc of growing medium.

The increasing levels of double acid extractable P, K and Ca at the initiation of bract color may again be attributed to their increasing supplies of the fertilizer solutions (Table XXVIII). Similarly, decreased levels of Ca and Mg are primarily the result of leaching.

To calculate the optimum value for each of these analytical methods, it is first essential to determine the level of optimum fertility. This has been described as the range where nutrient uptake levels off even though the supply of nutrients continues to increase (102).

Table XXV. Substrate analysis values as determined by the modified LSU method four weeks after the start of short days.<sup>1</sup>

Treatment	pH	P (ppm)	(meq/100 cc)		
			K	Ca	Mg
1. (0.25 X)	6.7	82	0.45	10.53	2.25
2. (0.5 X)	6.5	102	0.99	11.35	2.31
3. (1.0 X)	6.4	127	1.23	12.11	2.37
4. (1.5 X)	6.1	163	1.93	13.33	2.27
CV (%)	5	42	39	18	12

<sup>1</sup>Means of 12 observations.

Table XXVI. Substrate analysis values as determined by the modified LSU method at the initiation of bract color.<sup>1</sup>

Treatment	pH	P (ppm)	(meq/100 cc)		
			K	Ca	Mg
1. (0.25 X)	6.4	87	0.52	10.16	2.17
2. (0.5 X)	6.2	97	0.87	10.92	2.22
3. (1.0 X)	6.2	159	1.41	11.79	2.28
4. (1.5 X)	6.0	221	1.92	12.85	2.19
CV (%)	3	35	29	14	15

<sup>1</sup>Means of 12 observations.

Table XXVII. Substrate analysis values as determined by the modified Double Acid<sup>1</sup> method four weeks after the start of short days.<sup>2</sup>

Treatment	pH	P (ppm)	(meq/100 cc)		
			K	Ca	Mg
1. (0.25 X)	6.7	93	0.59	11.53	2.34
2. (0.5 X)	6.5	122	1.10	12.22	2.41
3. (1.0 X)	6.4	181	1.34	13.01	2.45
4. (1.5 X)	6.1	211	2.12	14.16	2.36
CV (%)	5	51	42	24	14

<sup>1</sup>Equal parts of 0.05 N HCl and 0.025 N H<sub>2</sub>SO<sub>4</sub>.

<sup>2</sup>Means of 12 observations.

Table XXVIII. Substrate analysis values as determined by the modified Double Acid<sup>1</sup> method at the initiation of bract color.<sup>1</sup>

Treatment	pH	P (ppm)	(meq/100 cc)		
			K	Ca	Mg
1. (0.25 X)	6.7	98	0.57	11.08	2.28
2. (0.5 X)	6.5	116	1.12	11.72	2.28
3. (1.0 X)	6.4	149	1.36	12.51	2.37
4. (1.5 X)	6.1	185	2.07	13.79	2.33
CV (%)	5	36	32	19	17

<sup>1</sup>Equal parts of 0.05 N HCl and 0.025 N H<sub>2</sub>SO<sub>4</sub>.

<sup>2</sup>Means of 12 observations.

However, this concept must be modified in the determination of optimum fertility levels for most containerized floricultural crops.

Constant fertilization programs and the limited volume of a container result in increased concentrations of soluble salts in the growing medium. If the supply of nutrients is increased, uptake will increase until the accumulation of these salts results in injury. Once this injury becomes severe enough to limit absorption, uptake becomes independent of supply. Therefore, it is apparent that optimum fertility does not occur within the range where nutrient uptake reaches equilibrium with nutrient supply.

Several factors must be considered in the determination of optimum fertility. Since floricultural crops are largely evaluated for their aesthetic characteristics, the qualitative response to fertility must be considered. Increased levels of fertility may be justified only if the quality of the plant is enhanced. However, any such improvement in quality must be significant enough to offset the danger from the increased potential of injury from soluble salts. Distinguishing these optimums becomes increasingly difficult as fertility rises and qualitative response becomes less conspicuous. Since no improvement in plant quality was noted at the increased (1.5X) level of fertility, it was concluded that the previously recommended (1.0X) fertility rate represented optimum.

Another factor to be considered in determining optimum

fertility is the stage of plant development. Tayama (224) has shown that from four weeks after the start of short days until finish, poinsettias approximately double in size. During this phase of active growth nutrients are extensively translocated throughout the plant. This results in a diluting effect on absorbed fertilizer salts. However, at the initiation of bract color, the development of new tissue is reduced and the accumulation of these salts may result (67). Therefore, the increase in nutrient uptake which occurred at the initiation of bract color represents the effect of luxury consumption and not optimum fertility. Based on these relationships, the probable optimum range for each of the three analytical methods was calculated by placing a 95 percent confidence interval around the (1.0X) treatment mean, four weeks after the start of short days (Table XXIX).

Several studies (243, 144, 43) have reported the recommended range of soil test values for floricultural crops based on HOAc extraction. These reports indicate an optimum P level of from 4-6 ppm. These values are considerably lower than those observed in this study. However, such differences may be attributed to the continuous supply of P provided in the fertilization program. Another important factor to be considered is the difference in the physical properties of soils and soilless growing media. The increased porosity and water-holding capacity associated with the bark medium provides a greater potential for nutrient retention. Therefore, increased P levels

Table XXIX. Interval estimate for the nutritional status of *Euphorbia pulcherrima* Willd. cv 'Annette Hegg Dark Red' based on three modified methods of substrate analysis.

Element	Extractant		
	Modified Spurway	Modified LSU	Modified double acid
P	19-50 (ppm)	105-150 (ppm)	163-200 (ppm)
K	30-42 (ppm)	1.16-1.31 (meq/100 cc)	1.24-1.44 (meq/100 cc)
Ca	113-184 (ppm)	11.52-12.71 (meq/100 cc)	12.43-13.60 (meq/100 cc)
Mg	24-49 (ppm)	1.88-2.87 (meq/100 cc)	1.91-2.99 (meq/100 cc)

<sup>1</sup>Calculated as a 95% confidence interval around mean values at the 1.0x level of fertility.



may be expected under these conditions.

The range for HOAc extractable K, Ca and Mg, as determined by this study, agreed well with previous recommendations for greenhouse crops (144). However, these results give no indication of the interactions between these elements. Therefore, interpretation of the analytical values is difficult.

Mastalerz (144) has recommended a P range of from 126-423 ppm for floricultural crops, based on extraction with the Bray solution. This agrees very well with the optimum values determined by this experiment.

White (243) has recommended a K level, based on  $\text{NH}_4\text{OAc}$  extraction, of from 0.76-1.50 meq/100 g. These values are difficult to relate to bark media because of differences in bulk density. However, it may be concluded that the optimum K levels, as determined by this study, exceed White's recommendation. This increased level may be attributed to the relatively high concentration of K used in the constant fertilization program.

Mastalerz's (144) recommendation for Ca and Mg, based on  $\text{NH}_4\text{OAc}$  extraction, ranges from 8.0-13.0 and 1.3-3.5 meq/100 g, respectively. Although these values are, again, difficult to relate to the bark medium, it may be concluded that the values determined by this experiment fell within these recommended ranges.

Few recommendations have been given for floricultural crops based on double acid extraction (151). Generally, these values are proportionately higher than those obtained

by extraction with  $\text{NH}_4\text{OAc}$ . This demonstrates the effect of the strong oxidizing and reducing acids used in the composition of the double acid solution.

Exchange extractants such as  $\text{NH}_4\text{OAc}$  and the double acid solution provide a good indication of the balance of nutrients in the growing medium. This is particularly evident with regard to the relationship between K, Ca and Mg. Shanks and Link (207) have reported greater Mg uptake associated with increased levels of applied N and K. However, the relationship between these nutrients may be significantly affected when in continuous supply.

Substrate analysis showed that the percentage of K saturation of the exchange capacity increased as the concentration of K increased in the fertilizer solutions. However, the percent saturation of Ca and Mg decreased even though the level of exchangeable Ca and Mg increased. Since K was in continuous supply the uptake of Ca and Mg was enhanced. However, as the level of K fertilization was increased, the K:Ca and K:Mg ratios became out of balance. This resulted in K antagonisms and reduced Ca and Mg uptake. Such changes in the relationships between nutrients emphasize the effect of fertility regimes on the interpretation of analytical values.

## CONCLUSIONS

The determination of the physical characteristics of southern pine bark indicate suitable drainage, aeration and water-holding capacities for use as a container medium. However, the bulk density of pine bark was shown to be considerably lower than that of field soils. Therefore, analytical samples must be adjusted for differences in weight and volume. Further adjustments in particle size distribution may also be required to increase sample homogeneity.

The total mineral analysis (from ash) of southern pine bark was determined as a background for the interpretation of substrate fertility. In general the concentrations of N, P, K, Ca, Mg, Fe, Zn and Mn fell within acceptable ranges. However, it was found that supplemental application of these elements would be necessary.

The three modified analytical systems used for the determination of nutrient "availability" differed in the quantity of nutrients extracted. Because HOAc removes only ions from the bulk solution, it yielded values less than the two exchange extractants tested. However, each system was found most effective at a 1:4 v/v substrate:solution

ratio, when extracted for 24 hours.

Nutrient "availability" is also influenced by the C:N ratio of a growing medium. Although the C:N of pine bark was found to be approximately 123:1, it was concluded that high levels of fertility and the short growing season for most floricultural crops off-set reductions in nutrient "availability."

Buffered  $\text{NH}_4\text{OAc}$  and  $\text{BaCl}_2$  were both found acceptable as index solutions for the determination of the CEC of pine bark. However, these two solutions were found most effective at a 1:4 v/v substrate:solution ratio, when extracted for 24 hours. In general, CEC values were approximately 13 meq/100 cc of bark.

Water and  $\text{CaCl}_2$  were evaluated for their effectiveness in estimating the pH of pine bark. Although water may be used,  $\text{CaCl}_2$  was found to provide the best estimate of pH. This may be attributed to the presence of excess soluble salts in bark. The use of a 1:4 v/v substrate:extractant ratio was also shown to reduce junction potential over narrower dilutions.

The selection of an optimum level of fertility for Euphorbia pulcherrima Willd. cv 'Annette Hegg Dark Red' was determined both quantitatively and qualitatively. The corresponding substrate analysis values from each of the three modified analytical methods were then determined. The Spurway test simulates the natural soil solution process. However, the LSU and Double Acid tests

utilize index ions to replace those on the exchange sites. Based on these data it may be concluded that each of the three methods provide an acceptable means of evaluating substrate fertility.

Pine bark became a popular source of soilless growing media because it was inexpensive and readily available. However, industry is continuously finding more profitable uses for wood by-products. This has resulted in the reduced availability and increased cost of raw bark. When such conditions occur growers naturally turn to alternate sources of growing media. Therefore, further adaptations of analytical methods will be required to handle those characteristics specific to new media sources.

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## VITA

Don C. Wilkerson was born on November 6, 1951, in St. Louis, Missouri. He attended McCluer Senior High School in Florissant, Missouri, where he graduated in 1969. In August of 1976 he completed the requirements for the B.S. degree in agriculture at Arkansas State University. He entered the University of Arkansas in September of 1976 and completed the requirements for the M.S. degree in horticulture in 1978. The title of his thesis was "Fluoride Toxicity in Chlorophytum comosum." After teaching for a year at the University of Southwestern Louisiana he entered Louisiana State University where he is currently a candidate for the Doctor of Philosophy degree.

## EXAMINATION AND THESIS REPORT

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Major Field: Horticulture

Title of Thesis: Some Physical and Chemical Properties of Pine Bark Growing Medium  
used as an Evaluation of its Nutritional Status

Approved:

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Date of Examination:

August 25, 1981